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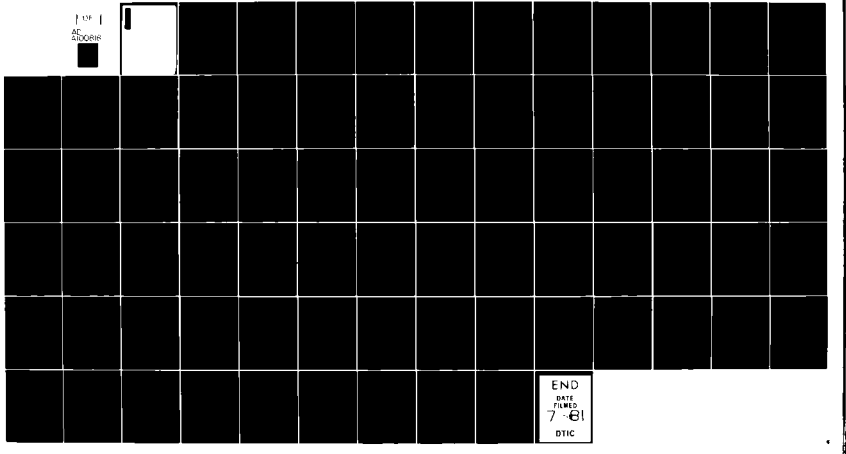

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CRYSTALLIZATION KINETICS OF TWO METALLIC GLASSES BY MOSSBAUER SPECTROSCOPY

Thesis

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CRYSTALLIZATION KINETICS OF TWO METALLIC GLASSES BY MOSSBAUER SPECTROSCOPY

THESIS

Presented to the Faculty of the School of Engineering of
of the Air Force Institute of Technology
Air University
In Partial Fulfillment of the
Requirements for the Degree of
Master of Science

by

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Graduate Nuclear Engineering

March 1981

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Preface

This thesis describes my efforts to determine the crystallization kinetics of two amorphous iron alloys, $Fe_{80}B_{20}$ and $Fe_{80}P_{6.5}C_{3.5}B_{10}$. The objectives of this study were: 1) to anneal the glasses at various temperatures, 2) to take Mossbauer spectra during the annealing, 3) to analyze the spectra to determine the growth of crystals, and 4) to use the crystallization rates to calculate the activation energy and projected lifetimes at 473 K. This thesis will summarize past work in this area, describe the equipment and methods, and present analysis of the Mossbauer spectra and the results, conclusions and recommendations.

I thank Dr. Harold Gegel of the Air Force Materials Laboratory for sponsoring this study and supplying the glassy metal ribbons. I especially thank my advisor, Dr. George John, for his continuous support and guidance. Finally, I am grateful to my wife, Judy, and my sons, David and Timothy, for their constant devotion and support during this long study.

Contents

		Page
Preface		ii
List of Figures		v
List of Tables		vii
Abstract		viii
I.	Introduction	1
	Background	1
	Problem	2
	Scope	2
	Review of the Literature	2
	Assumptions	5
	Overview	5
II.	Theory	6
III.	Equipment and Procedures	10
	Mossbauer Equipment	10
	Annealing System	10
	Sample Preparation	13
	Heater Assembly	13
	Annealing and Data Collection	14
	Data Processing	15
	Subroutine CALFUN (Gaussian fit to $Fe_{80}B_{20}$ glass)	15
	Subroutine CALFUN (Crystallized $Fe_{80}B_{20}$)	16
	Subroutine CALFUN (for α-Fe, peaks one and six)	17
	Goodness of Fit	18
IV.	Results and Discussion	19
	Discussion	36
V.	Conclusions and Recommendations	40
	Recommendations	40

Page

Bibliography. 42

APPENDIX A: GAUSSCALF. 44

APPENDIX B: FIVECALF 48

APPENDIX C: ALPHA-BG 54

APPENDIX D: GENFIT Instructions. 58

APPENDIX E: Applications of Metallic Glasses 62

Vita. 65

List of Figures

Figure		Page
1	Heater	11
2	Mossbauer/Annealing System	12
3	Sample/Heater Assembly	14
4	Mossbauer Spectrum of $Fe_{80}B_{20}$, 573 K, 2.08 Hr, GAUSSCALF, x = 0.	21
5	Mossbauer Spectrum of $Fe_{80}B_{20}$, 573 K, 48.9 Hr, GAUSSCALF, x = 0	22
6	Mossbauer Spectrum of $Fe_{80}B_{20}$, 573 K, 111 Hr, FIVECALF, x = 0.0512.	23
7	Mossbauer Spectrum of $Fe_{80}B_{20}$, 573 K, 281 Hr, FIVECALF, x = 0.190	24
8	Mossbauer Spectrum of $Fe_{80}B_{20}$, 573 K, Fully Crystallized, FIVECALF.	25
9	Mossbauer Spectrum of $Fe_{80}B_{20}$, 611 K, Fully Crystallized, FIVECALF.	26
10	Mossbauer Spectrum of $Fe_{80}B_{20}$, 611 K, Fully Crystallized, FIVECALF with Quadrupole Splitting.	27
11	Mossbauer Spectrum of $Fe_{80}B_{20}$, 611 K, Run 3 Fully Crystallized, FIVECALF.	28
12	Mossbauer Spectrum of $Fe_{80}B_{20}$, 604 K, 13.8 Hr, FIVECALF, x = 0.316	29
13	Mossbauer Spectrum of $Fe_{80}B_{20}$, 604 K, Fully Crystallized, FIVECALF.	30

Figure		Page
14	Mossbauer Spectrum of $Fe_{80}P_{6.5}C_{3.5}B_{10}$, 716 K, 2.87 Hr, ALPHA-BG, x ≅ 1	31
15	Mossbauer Spectrum of $Fe_{80}P_{6.5}C_{3.5}B_{10}$, 716 K, Fully Crystallized, ALPHA-BG	32
16	Crystallized Fraction x(t) vs Time.	34
17	Arrhenius Plot.	35

List of Tables

Table		Page
I	Annealing Runs	20

Abstract

In this study, Mossbauer spectroscopy was used to examine thermal aging of two metallic glasses. $Fe_{80}B_{20}$ was isothermally annealed at 573, 604, 611, and 626 K; and $Fe_{80}P_{6.5}C_{3.5}B_{10}$ was annealed at 614, 716, and 744 K. The activation energy of $Fe_{80}B_{20}$, determined from the growth of α-Fe crystals, was 0.256 ± 0.006 MJ/mole. The projected lifetime of this glass, based on the onset of crystallization, is 400 years. No quantitative data were obtained for $Fe_{80}P_{6.5}C_{3.5}B_{10}$; however, based on the higher temperature required for crystallization, it is expected to have a longer lifetime.

CRYSTALLIZATION KINETICS OF TWO METALLIC GLASSES BY MOSSBAUER SPECTROSCOPY

I. Introduction

Mossbauer spectroscopy is becoming an increasingly important tool for studying the environments of nuclei. In this study, it was used to examine the crystallization characteristics of $Fe_{80}B_{20}$ and $Fe_{80}P_{6.5}C_{3.5}B_{10}$ amorphous alloys. These materials, commonly called metallic glasses, crystallize during accelerated aging at high temperatures.

Background

The glassy metals exhibit useful magnetic, as well as material (tensile strength, hardness, flexibility), properties.[1] The Air Force Materials Laboratory has become interested in their possible use in magnetic devices for Air Force weapons systems. As a result of this interest, in 1978 Schmidt (Ref 1) and Roberts (Ref 2) used Mossbauer spectroscopy to study the atomic structure of a few of the glassy metals, including $Fe_{80}B_{20}$. Because of projected high-temperature applications, the Materials Laboratory is concerned about thermal aging of these materials. Knowledge of the glasses' expected lifetimes at operating temperatures around 473 K is needed. To predict the aging rates of these amorphous materials, one must know

[1] See Appendix E for a review of possible applications.

their crystallization characteristics. Many methods have been used to determine these rates; all require measurements at 100 to 200 K above the expected operating temperature. In this study, Mossbauer spectroscopy was used to examine the growth of crystals in the metallic glasses $Fe_{80}B_{20}$ and $Fe_{80}P_{6.5}C_{3.5}B_{10}$.

Problem

The problem investigated in this study was to determine the thermal aging rate of metallic glasses. Specifically, the kinetics of crystallization of $Fe_{80}B_{20}$ and $Fe_{80}P_{6.5}C_{3.5}B_{10}$ were studied. These glasses were examined by: 1) isothermal annealing, 2) taking Mossbauer spectroscopy during annealing, 3) evaluating the spectra to determine growth of α-Fe crystals, and 4) using the crystallization rates at various temperatures to determine the Arrhenius constant.

Scope

This study was limited to the investigation of only two of the glassy metals, $Fe_{80}B_{20}$ and $Fe_{80}P_{6.5}C_{3.5}B_{10}$. The temperature ranges were respectively 573 to 626 K and 716 to 744 K. The annealing periods were from one day at the high temperatures to two weeks at the lowest temperatures. No attempt was made to determine the structure of either the amorphous or the crystalline material. In addition, the literature studied was limited to only sources available at the School of Engineering, Air Force Institute of Technology.

Review of the Literature

This section contains a review of some of the many studies of the metallic glasses. These studies are mainly

concerned with three characteristics of the glasses: 1) amorphous structure, 2) temperature dependence of their magnetic properties, and 3) thermal aging (or crystallization) characteristics. Some of the results of these studies will be presented in the discussion section in Chapter IV.

Luborsky studied the crystallization of $Fe_{80}B_{20}$, $Fe_{40}Ni_{40}P_{14}B_6$, and $Fe_{40}Ni_{40}B_{20}$, using magnetic methods and differential scanning calorimetry (Ref 3). Using the temperature of onset of crystallization at various heating rates, he determined the activation energy for the three materials. He also showed that thermal stability increased with the number of atomic species, i.e., that $Fe_{80}B_{20}$ was the least stable of the three glasses. Fukamichi and others studied the magnetization, electrical resistivity, thermal expansion, and differential thermal change of a variety of Fe-B glasses (Ref 4). They determined that the crystallization mechanism of the Fe-B glasses depended on the concentration of boron. Chien studied $Fe_{80}B_{20}$ from 4.2 K up to 1050 K, using Mossbauer spectroscopy, and found that it crystallized to α-Fe and Fe_3B when annealed at a high heating rate, but found only Fe_2B at low heating rates (Ref 5). Luborsky and Lieberman examined the crystallization kinetics of the Fe-B glasses (12 to 28 percent boron) by differential scanning calorimetry (Ref 6). They determined that for 18 to 28 percent boron, the activation energy for the onset of crystallization was independent of boron concentration. However, Tarnoczi and others studied the role of Fe_3B in the crystallization of Fe-B glasses, and concluded

that $Fe_{80}B_{20}$ was most stable (Ref 7). Matsuura, in a study similar to Luborsky and Lieberman's above, used differential thermal analysis on 12 to 20 percent boron Fe-B glasses (Ref 8). His results, obtained above 700 K, led to the conclusion that the formation of α-Fe accompanies the crystallization of Fe_3B. Chien and others did a lengthy study on Fe-B glasses (14 to 28 percent boron) and crystalline Fe_3B using Mossbauer spectroscopy and magnetization measurements (Ref 9). Kemeny and others used Mossbauer spectroscopy, differential scanning calorimetry, and magnetization measurements for a thorough investigation of the structure and crystallization of Fe-B metallic glasses (12 to 25 percent boron) (Ref 10). For 16 to 25 percent boron, they concluded that crystallization proceeds by the formation of α-Fe and Fe_3B in an eutectic process. They also deduced that the glass structure should be based on locally distorted, quasi-crystalline Fe_3B. Schaafsma and others have also done a lengthy crystallization study on two $Fe_{80}B_{20}$ glasses (Ref 11). They concluded that the crystallization mechanism did not change between 580 and 640 K. They also found that nucleation did not control the crystallization rate; i.e., that crystal nuclei exist in the as-quenched amorphous material. Finally, Kopcewicz used Mossbauer spectroscopy to study radio-frequency annealing of $Fe_{40}Ni_{40}B_{20}$ (Ref 12). He found that as a result of magnetostrictively induced atomic vibrations, a strong rf field caused crystallization in LN_2- cooled samples.

Assumptions

The following were assumed to be true at the outset:

1) The $Fe_{80}B_{20}$ and $Fe_{80}P_{6.5}C_{3.5}B_{10}$ alloys were amorphous.
2) During annealing α-Fe and metastable Fe_3B were formed.
3) The compositions were true to within one percent.

Overview

The theory of the Mossbauer effect has been well documented and will not be presented here; however, the use of Mossbauer spectroscopy to study nuclear environments is presented in Chapter II, along with the theory of crystallization of Fe-B glasses. The Mossbauer equipment, annealing system, experimental procedures, and data processing are described in Chapter III. Chapter IV contains the results and discussion, and Chapter V contains the conclusions and recommendations.

II. Theory

The theory of the Mossbauer effect has been fully developed and is well understood (see, e.g., Ref 13 and 14). For a condensed and simplified explanation, see Roberts' thesis (Ref 2: 3-11). Mossbauer spectroscopy measures the hyperfine fields of nuclei. This is possible because the hyperfine field interacts with the nuclear dipole moment, which, for iron-57, splits the resonant-absorption energy into six energy levels. The magnitudes of these energies are directly proportional to the value of the hyperfine field, which is characteristic of the electron environment of the nucleus. Thus, for nuclei in a crystal, there are different six-peak spectra (with Lorentzian line shapes) for each magnetically inequivalent site. For an amorphous material which has only short-range order, there are very few magnetically equivalent sites. The glass spectrum, then, is a combination of many different six-peak spectra, with a probability distribution P(H) describing the nuclear hyperfine fields. The P(H) of $Fe_{80}B_{20}$ have been described by Schmidt (Ref 1) and Vincze (Ref 15) as a binomial distribution. This distribution was related to the number of nearest neighbors of iron and non-iron nuclei. However, others have shown that the glass spectra can be described by a model-independent probability distribution (Refs 15, 16, and 17). Schurer and

and Morrish have shown that a single six-line pattern, using Gaussian line shapes, describes the glass spectrum reasonably well (Ref 16: 819). This method is used in this study for the $Fe_{80}B_{20}$ glass.

When a metastable metallic glass is heated, it undergoes atomic rearrangement to a more stable, but still amorphous, state (Ref 18: 577). With further heating, it begins to crystallize. This crystallization has been described as a diffusion process, as one or more species migrates out of the amorphous region (Ref 11: 4428). For the Fe-B glasses, the possible crystalline states are α-Fe, FeB, Fe_2B, and Fe_3B. Fe_2B is expected to form during crystallization of the amorphous phase because it is much more stable than Fe_3B. For $Fe_{80}B_{20}$, however, the metastable Fe_3B (or $Fe_{75}B_{25}$) is much nearer the original composition than Fe_2B (or $Fe_{67}B_{33}$). Thus, others have found that α-Fe and Fe_3B are the species formed during annealing of $Fe_{80}B_{20}$ (Refs 7: 1026; 8:232; 10: 485; 11: 4429). While some determined that iron diffused from the glass to form α-Fe, leaving amorphous $Fe_{75}B_{25}$, which subsequently crystallized to Fe_3B (Ref 11: 4429), others deduced that Fe_3B crystallized accompanied by the simultaneous formation of α-Fe (Ref 8: 233). For a glass with many species, such as $Fe_{80}P_{6.5}C_{3.5}B_{10}$, there are many possible crystalline states. These include FeB, Fe_2B, Fe_3B, Fe_3C, Fe_3P, and combinations of these by atomic substitution, alone with α-Fe.

The isothermal crystallization of an iron-based glass can be followed, then, by examining the growth of the six-peak

α-Fe spectrum in a series of Mossbauer spectra. The crystalline fraction x(t) is the ratio of the volume of α-Fe crystals formed at time t to the volume in the fully crystallized sample. It can be described by the Johnson-Mehl-Avrami equation (Ref 11: 4426):

$$x(t) = 1 - \exp\left[-(k(T)t)^n\right] \qquad (1)$$

When rearranged, Eq (1) becomes

$$\ln \ln \frac{1}{1-x(t)} = n \ln k(T) + n \ln t \qquad (2)$$

which yields a straight line plot with slope n. Above, k(T) is a temperature dependent constant, and the exponent n is determined by the nucleation and growth characteristics of the crystallization. The time $t_x(T)$ to crystallized fraction x can be determined from Eq (2) for a series of isothermal measurements. Then, for a thermally-activated diffusion process, the Arrhenius equation describes the lifetime t_x at constant x as a function of temperature T:

$$t_x = k_0^{-1} \exp\left(E_A/k_B T\right) \qquad (3)$$

where k_0 is a frequency factor, k_B is the Boltzman constant, and E_A is the activation energy of the crystallization process expressed as energy per atom (or per mole) (Ref 11: 4427). Eq (3), when rearranged, also yields a straight line plot, with slope E_A/k_B:

$$\ln t_x = \ln k_0^{-1} + (E_A/k_B)T^{-1} \qquad (4)$$

The above kinetic parameters can be derived from relatively quick measurements at high temperatures. If the assumption is valid that the crystallization process is the same over a large range of temperatures, then these constants can be used to predict the crystallization rate at lower temperatures. In this study, the crystallized fractions x(t) were determined from the amplitudes of the α-Fe portions of Mossbauer spectra. The half lives $t_{.5}$ were then determined by least-squares curve fitting, and were used to determine the activation energy from the Arrhenius plot.

III. Equipment and Procedures

In this section the Mossbauer spectroscopy equipment and annealing system are described. The procedures for preparing the glass samples and assembling them in the heater are included. Annealing the samples, taking the Mossbauer spectra during annealing, and analyzing the spectra are also described.

Mossbauer Equipment

The major Mossbauer spectroscopy components included a constant-acceleration velocity transducer (motor), a linear amplifier/single channel analyzer, a krypton-filled proportional counter, and a Mossbauer control unit (MCU). All were manufactured by Ranger Electronics. The Mossbauer spectrum was taken on an RIDL 400 multichannel analyzer (MCA), operated in the time-sequential scaling mode. With the exception of the time-base oscillator, which was an RIDL model 54-6, the equipment is the same as that described by Skluzacek (Ref 19: 5-11), Schmidt (Ref 1: 11-15), and Roberts (Ref 2: 12). The source, connected directly to the motor, was approximately 6 mCi cobalt-57 in a rhodium foil.

Annealing System

The annealing system consisted of a heater, a thermocouple reader, a vacuum system, and one of two temperature controllers. Ranger Engineering built the heater (Fig 1) as

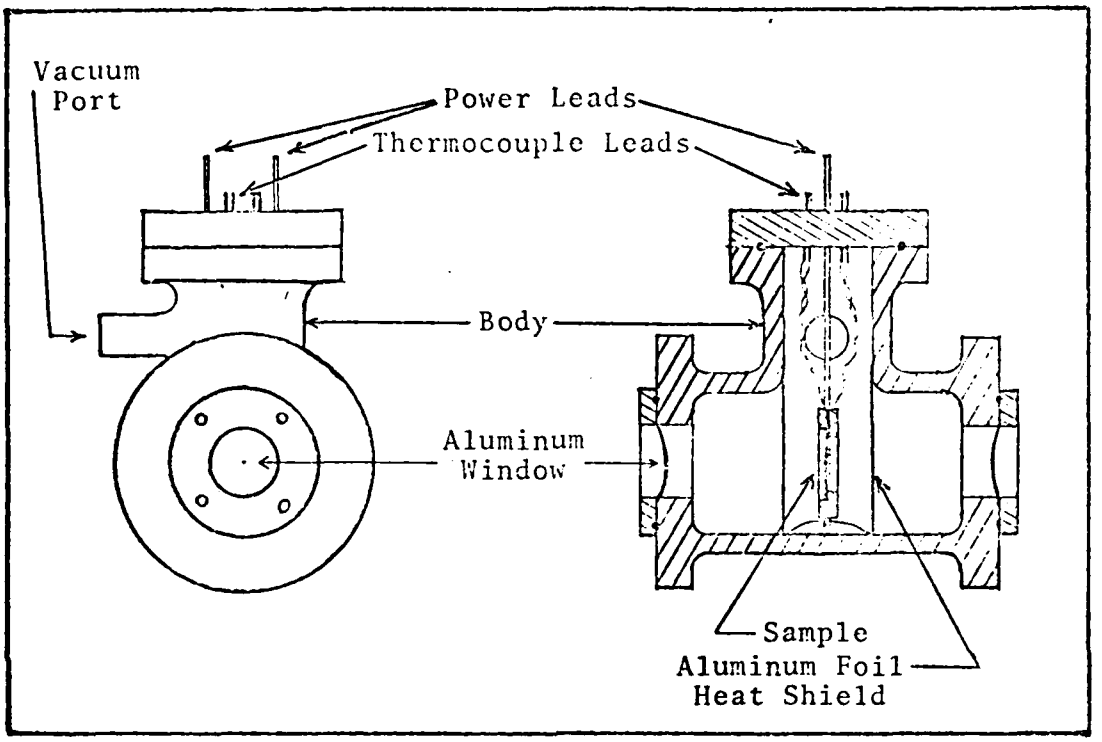

Fig 1. Heater (not to scale)

a prototype. The heating elements were two graphite discs (0.135 ± 0.002 mm x 25.4 mm). The sample was held between the heating discs and aligned with two aluminum foil windows (0.025 ± 0.001 mm x 25.4 mm). There were two iron-constantan thermocouples inside the heater; one was located in a slot at the edge of the heating element frame, the other was centered on the sample. The Omega thermocouple reader was calibrated with ice, boiling water, and molten tin. The vacuum system consisted of a forepump and an oil diffusion pump trapped with liquid nitrogen. Its purpose was to prevent convective heat transfer to the heater body. The first temperature controller,

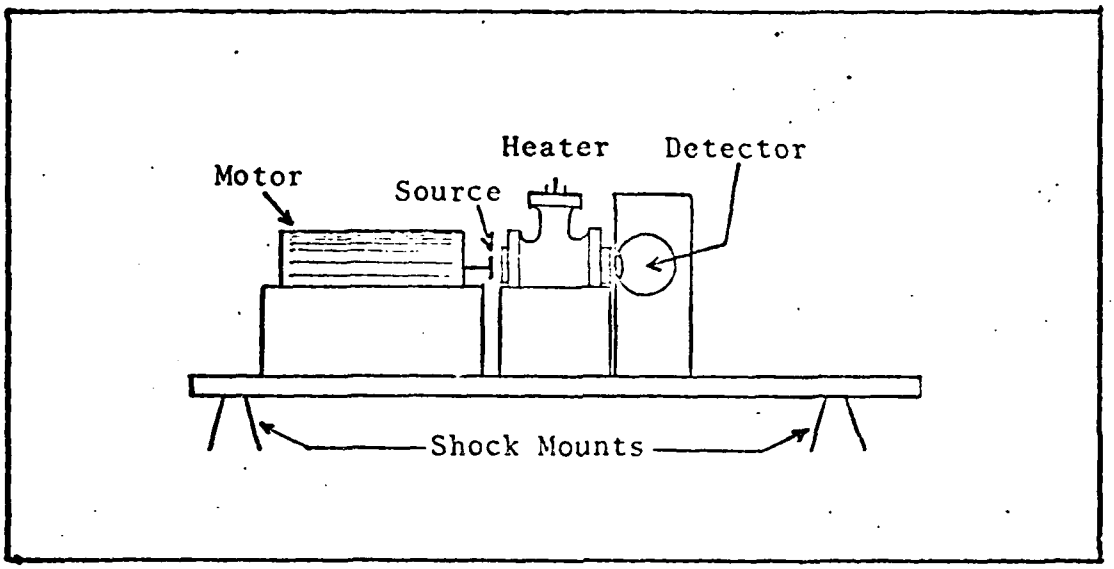

Fig 2. Mossbauer/Annealing System

a Lambda regulated DC power supply, was designed to provide manually-set constant current. Because of the failure of this power source, it was replaced with a Gardsman temperature controller, which was connected through a filament transformer to the heater. It supplied AC power, which produced an alternating field between the heating elements. This produced a problem which is discussed in Chapter IV. The edge thermocouple supplied feedback to the controller. Neither controller regulated the temperature adequately; temperatures varied ±2 K throughout the runs, with occasional drops (less than 1% of annealing time) of up to 10 K.

A sketch of the source-absorber-detector is shown in Fig 2. The distance from the source to the detector window was approximately 10 cm.

Sample Preparation

Dr. Harold Gegel, of the Air Force Materials Laboratory, provided the glassy metal ribbons which were used to prepare the samples. Battelle Laboratories at Columbus, Ohio, manufactured the ribbons by spin-cooling the molten alloy on a cooled rotating drum. The $Fe_{80}B_{20}$ (nominal atom percent) glass ribbons were 28.8 ± 0.5 μm thick, the $Fe_{80}P_{6.5}C_{3.5}B_{10}$ ribbons were 27.9 ± 0.5 μm thick, and the width of both varied from 0.5 to 1.2 mm with an average width of about 0.8 mm. The samples were prepared as a parallel array of the ribbons in 32 ± 2 mm long strips, to form an absorber 25 mm wide. The strips were held parallel with cellophane tape at the top and bottom and then bonded at one end with cyanoacrylate cement to a boron nitride disc (25.4 mm diameter). This was done to prevent thermal stresses which have been observed to affect the spectra (Ref 20). When the cement had dried, the glass strips were trimmed to the dimension of the boron nitride disc. Since the cement degraded during the annealing, the strips were held unfettered and remained free of thermal stress.

Heater Assembly

The sample and heating discs were assembled into the heater as shown in Fig 3. One graphite disc was inserted into the ceramic frame, followed by a clean boron nitride disc. The fine thermocouple was then centered on the boron nitride disc. The second boron nitride disc, with the glass sample attached, was then inserted, followed by the second graphite disc. The assembly was secured by two small plates and screws.

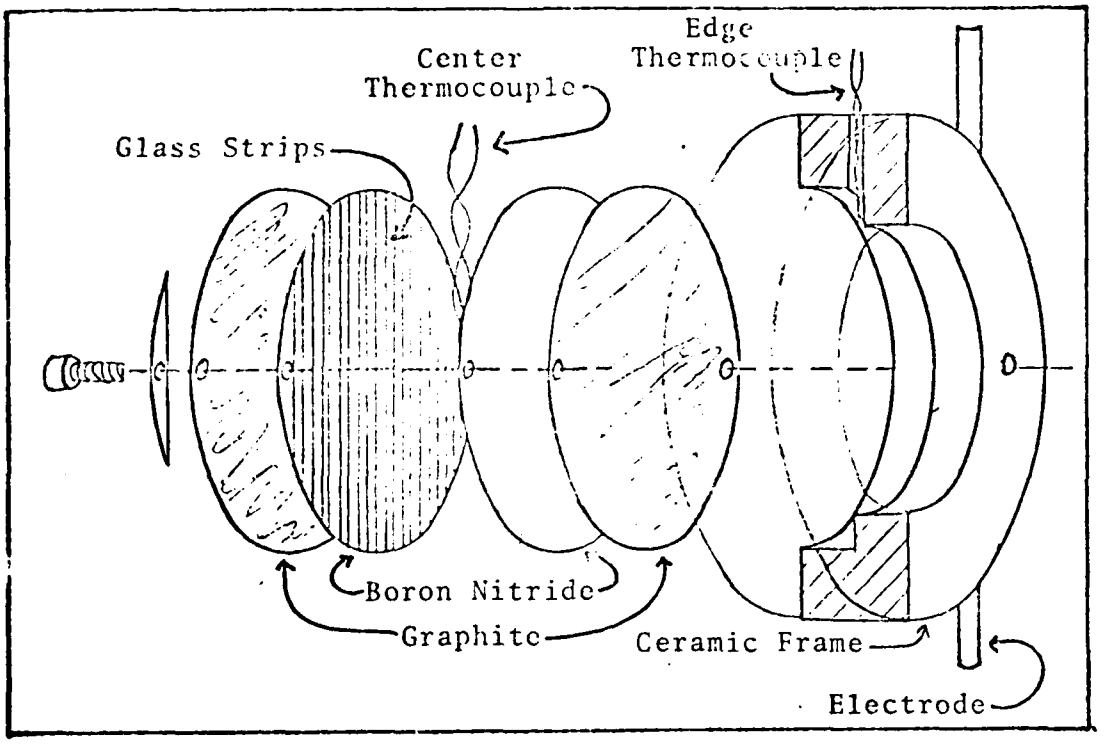

Fig 3. Sample/Heater Assembly

The heater assembly was then inserted into the heater body, secured, and vacuum was applied.

Annealing and Data Collection

Once the heater was assembled and degassed overnight, the isothermal annealing run began. The heating rate was 5 to 15 K/min up to 473 K, and held there until again degassed. When the vacuum dropped below 10^{-3} Pa, the temperature was increased to the desired annealing point. Generally, this took about five minutes. The initial Mossbauer spectrum was started as soon as this temperature was reached. Additional spectra were then taken in sequence until the sample was near full

crystallization. The time between the end of one spectrum and the beginning of the next was about ten minutes, which was the time necessary to punch the 400 channels of data onto paper tape. The isothermal annealing runs lasted from 10.5 hrs for $Fe_{80}B_{20}$ at 626 K, up to 315 hrs for $Fe_{80}B_{20}$ at 573 K. To fully crystallize the samples, the temperature was increased for about 18 hrs following each annealing run (to 640 K for $Fe_{80}B_{20}$, 750 K for $Fe_{80}P_{6.5}C_{3.5}B_{10}$). The temperature was then lowered to the original annealing point, and a final Mossbauer spectrum was taken. The spectrum collection periods were between 1 and 24 hrs, depending upon the crystallization rate of the sample.

Data Processing

The Mossbauer spectra were analyzed by a least-squares minimization curve fitting program developed at the Argonne National Laboratory. This program, GENFIT, has been modified by Skluzacek (Ref 1: 21), Schmidt (Ref 1: 21-22), and Roberts (Ref 2: 18). It is listed as Appendix A of Roberts' thesis (Ref 2: 50-60), and will not be repeated here. A user-supplied subroutine, CALFUN, provides the mathematical model to be fitted, along with the desired variable parameters. Three different CALFUNs were used for this study. They are explained below.

Subroutine CALFUN (Gaussian fit to $Fe_{80}B_{20}$ glass). This subroutine fits Gaussian line shapes to the $Fe_{80}B_{20}$ glass spectra taken at the beginning of each annealing run. Although

this method can lead to erroneous values of the average hyperfine field (Ref 10: 478), it did provide reasonable fits. Its purpose was to provide the glass spectra to be included in the CALFUN below. The variables which were included in this subroutine were:

1. Baseline: the average counts in the background of the Mossbauer spectrum.
2. Magnetic field: one value of average hyperfine field, in kOe (100 kOe = 7.96 MA/m).
3. Isomer shift: one value of average isomer shift for the glass, in mm/sec.
4. Quadrupole split: one value of average quadrupole split for the glass, in mm/sec.
5. Total intensity: one value for total intensity (average) of peaks one and six, expressed as a fraction.
6. Relative intensity: one value of the ratio of the average intensity of peaks two and five to the average intensity of peaks one and six.
7. Linewidths: six values of the full width at half maximum intensity (FWHM); one for each of the six Gaussian line shapes, in mm/sec.

The areal ratios of peaks three and four were constrained to one-third the area of peak one. This subroutine is listed as Appendix A. It was called GAUSSCALF.

Subroutine CALFUN (Crystallized $Fe_{80}B_{20}$). This subroutine provides for fitting the data with one six-peak α-Fe

spectrum, three six-peak Fe_3B spectra, and one six-peak (Gaussian) glass spectrum. The variable parameters are:

1. Baseline: as in GAUSSCALF above.
2. Magnetic fields: one hyperfine field for α-Fe and one each for three Fe_3B sites, in kOe.
3. Isomer shifts: one each for α-Fe and the three Fe_3B sites, in mm/sec.
4. Linewidths: one value of FWHM for peaks one and six, one for peaks two and five, and one for peaks three and four, of all three Fe_3B sites, in mm/sec.
5. Total intensities: one value of total intensity for each of α-Fe, Fe_3B, and the glass.

To simplify--and reduce the time and cost of--processing, this subroutine required many constraints. The glass spectrum was constrained to those parameters found in GAUSSCALF above, only its intensity was variable. The linewidth of α-Fe was constrained to that value found for the fully crystallized spectrum, and all six peaks used this same value. The areal ratios were 3:2:1:1:2:3 for peaks 1:2:3:4:5:6 of α-Fe and Fe_3B. The relative intensities of the three Fe_3B sites were 1:1:1, and their linewidths were constrained to be equal for similar peaks. Finally, the quadrupole splits of α-Fe and the three Fe_3B sites were constrained to zero. This subroutine, called [illegible]FITCALF, is listed as Appendix B.

Subroutine CALFUN (for α-Fe, peaks one and [illegible]. This version of CALFUN provides for analysis of only peaks one and six of the α-Fe crystallized from the glass. It includes a

Gaussian shaped background, and does not fit the center portion of the spectrum. The required variables are:

1. Baseline: as in GAUSSCALF.
2. Magnetic fields: one value for the α-Fe hyperfine field, and one value for the background, in kOe.
3. Total intensities: one value for the total intensity of peaks one and six of α-Fe, and one value for the background.
4. Linewidths: one value of FWHM for α-Fe, and one value for the background, in mm/sec.
5. Isomer shifts: one value of isomer shift for α-Fe and one value for the background, in mm/sec.

This subroutine is listed in Appendix C, and was called ALPHA-BG. Appendix D contains instructions for using GENFIT and CALFUN, and discusses required alterations to FIVECALF for spectra taken at other temperatures.

Goodness of Fit

The goodness of fit to the Mossbauer spectra is measured by Chi-squared, which is generated by GENFIT. Its value is defined by:

$$Chi^2 = \sum_{i=1}^{N} \frac{(\text{data point}_i - \text{calculated point}_i)^2}{\text{data point}_i} \qquad (5)$$

where N is the number of data points fitted. Theoretically, the values of Chi-squared obtained for a number of spectra should be randomly distributed around the number of data points, in the limit approaching this number.

IV. Results and Discussion

In this section, the results of the spectra analyses and crystallization rate determinations are presented. These results are compared in the discussion to those derived by others. Table I lists the isothermal annealing runs with the sample material, temperature, annealing period, slope of the Johnson-Mehl-Avrami plot, crystallization half-life $t_{.5}$, and heater power (AC or DC). Figures 4 through 15 are examples of the Mossbauer spectra taken during the annealing runs. The title of each figure lists the sample material, temperature, annealed time, CALFUN used, and crystalline fraction. The crystalline fraction was calculated as the ratio of the value of the α-Fe intensity of that run to the value of the fully crystallized state. Figures 4 (2.89 hr at 573 K) and 5 (48.9 hr at 573 K) show that the glass spectrum changes very little before crystallization. Figure 6 shows the α-Fe peaks just after the onset of crystallization; note that the remainder of the spectrum still resembles the glass spectra. Figures 7 and 8 (19 and 100 percent crystallized) include the locations of the peaks of the α-Fe and three Fe_3B spectra, along with the values of their hyperfine fields (in MA/m). Note that the values of the three Fe_3B fields increased with time, which was true of all isothermal annealing runs. However, the linewidths of these peaks decreased with time, as much as 50 percent. Figures 9 and 10

TABLE I

Annealing Runs

Run	Glass	Temperature (K ± 2 K)	Annealing Period (hr)	Half-life (hr)	Slope n	Power Type
1	$Fe_{80}B_{20}$	573	315	604 ± 57	1.60±0.22	DC
2	$Fe_{80}B_{20}$	604	45	34.0 ± .5	1.77±.24	AC
3	$Fe_{80}B_{20}$	611	76	18.0 ± 0.7	1.50±0.06	DC
4	$Fe_{80}B_{20}$	611*	48	21.1 ± 1.8	1.17±0.08	DC
5	$Fe_{80}B_{20}$	626	10.5	6.69 ± 0.54	1.57±0.14	DC
6	$Fe_{80}P_{6.5}C_{3.5}B_{10}$	614	27	No crystallization observed		AC
7	$Fe_{80}P_{6.5}C_{3.5}B_{10}$	716	48	<2.9		AC
8	$Fe_{80}P_{6.5}C_{3.5}B_{10}$	744	25	<0.5		AC

*This sample was annealed 13 days at 573 K, and was about 22% crystallized at the beginning of this run.

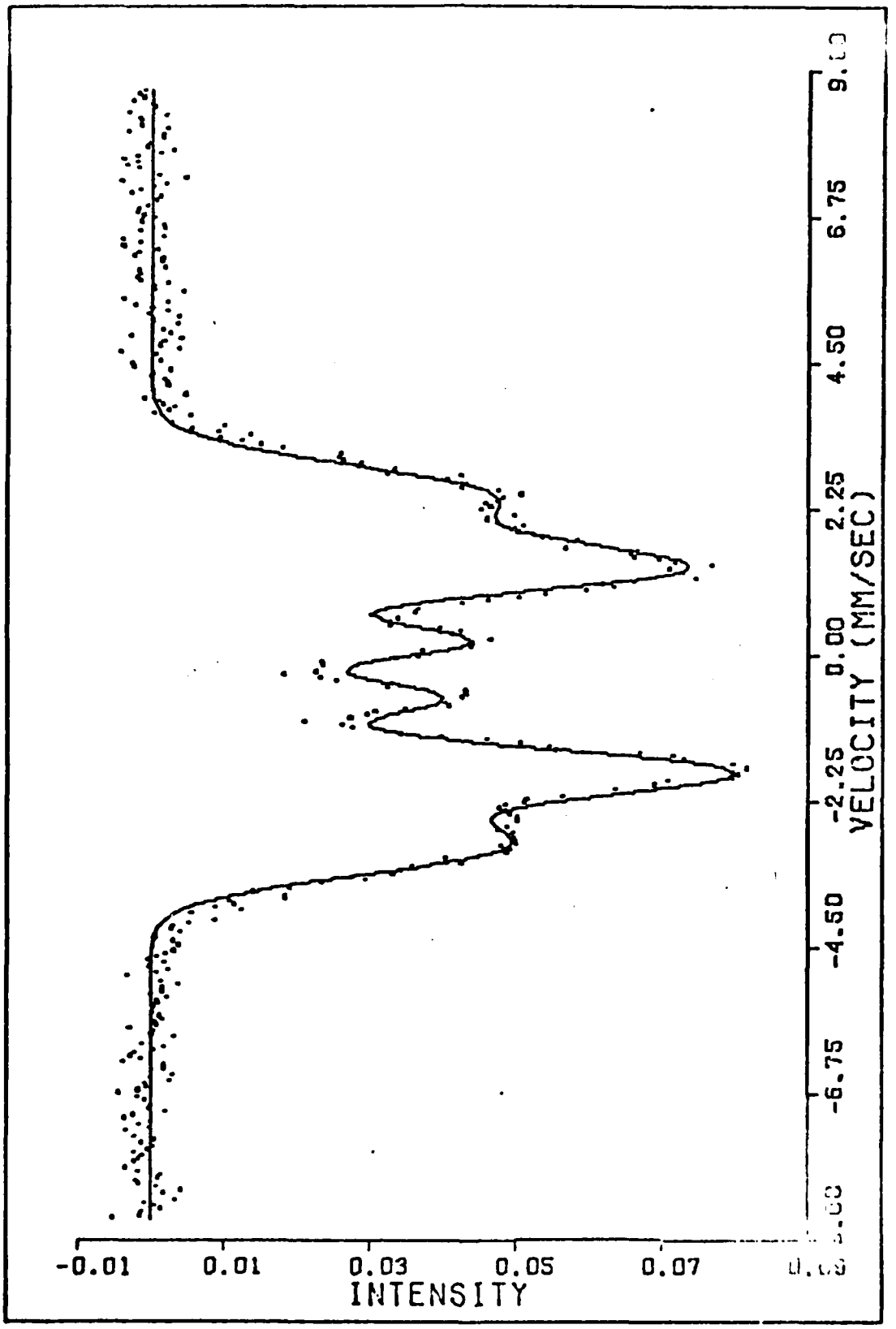

Fig. 4. Mossbauer Spectrum of $Fe_{80}B_{20}$, 573 K, 2.08 Hr, GAUSSCALF, x = 0

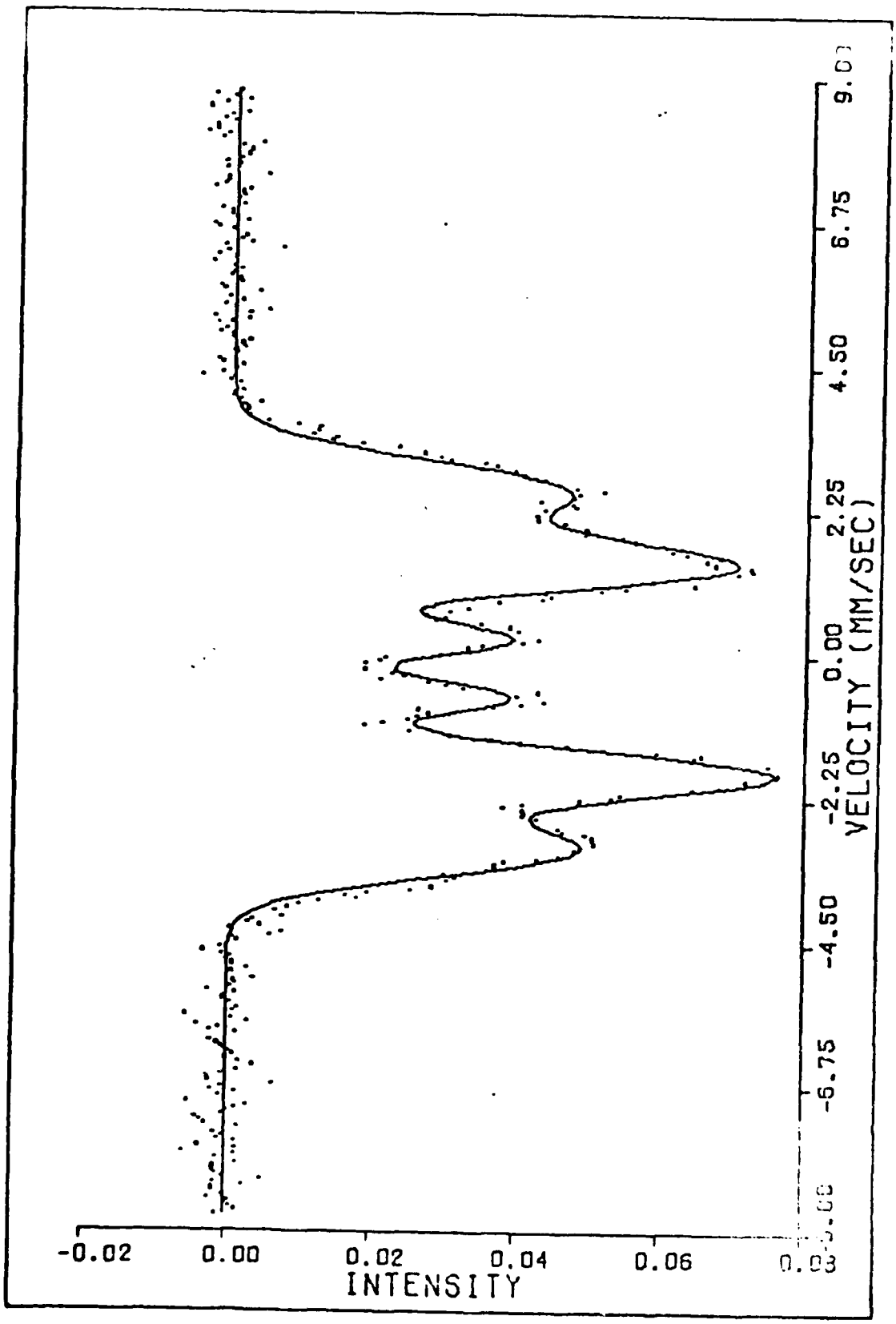

Fig. 5. Mossbauer Spectrum of $Fe_{80}B_{20}$, 573 K, 48.9 Hr, GAUSSCALF, x = 0

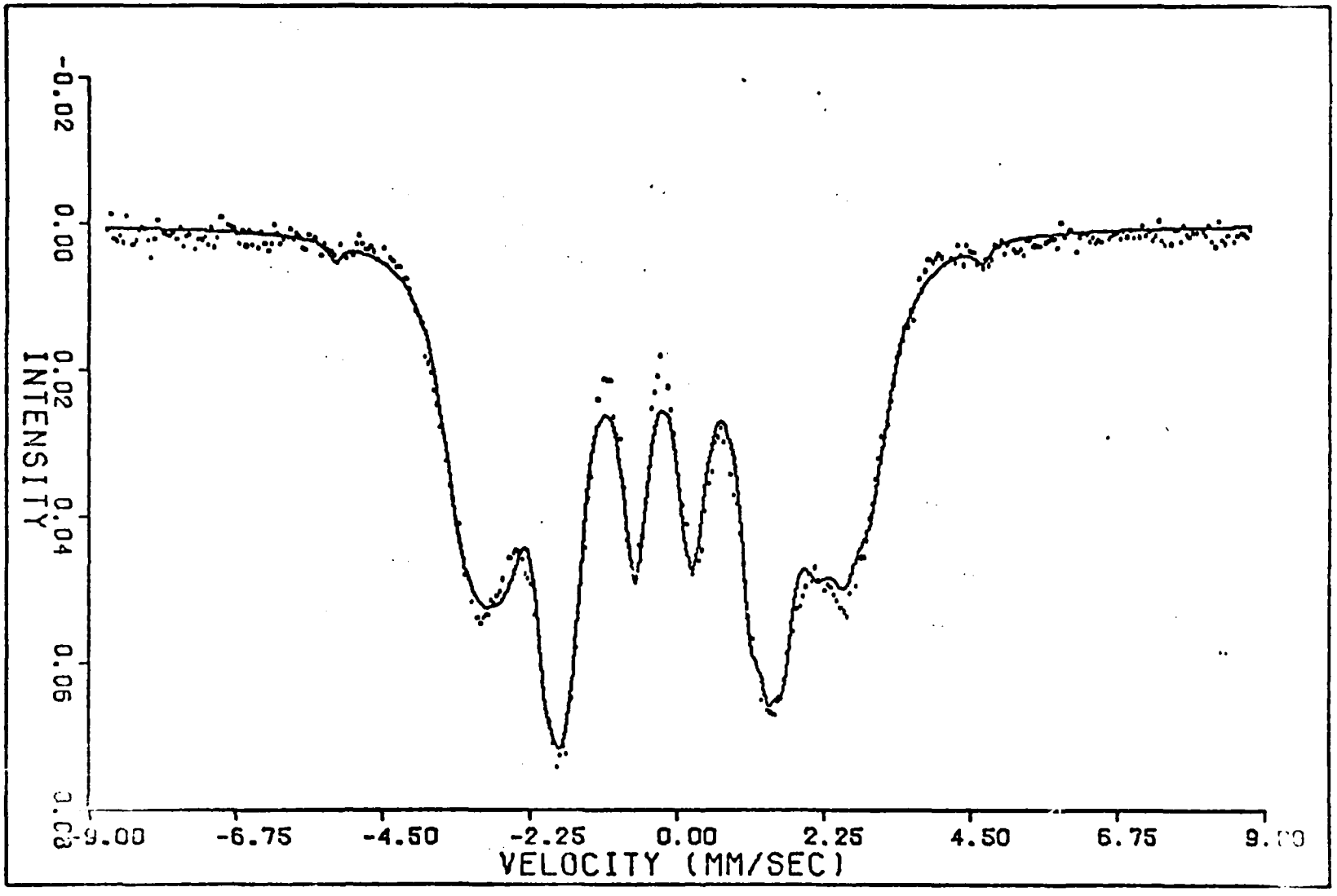

Fig 6. Mossbauer Spectrum of $Fe_{80}B_{20}$, 573 K, 111 Hr, FIVECALF, x = 0.0512

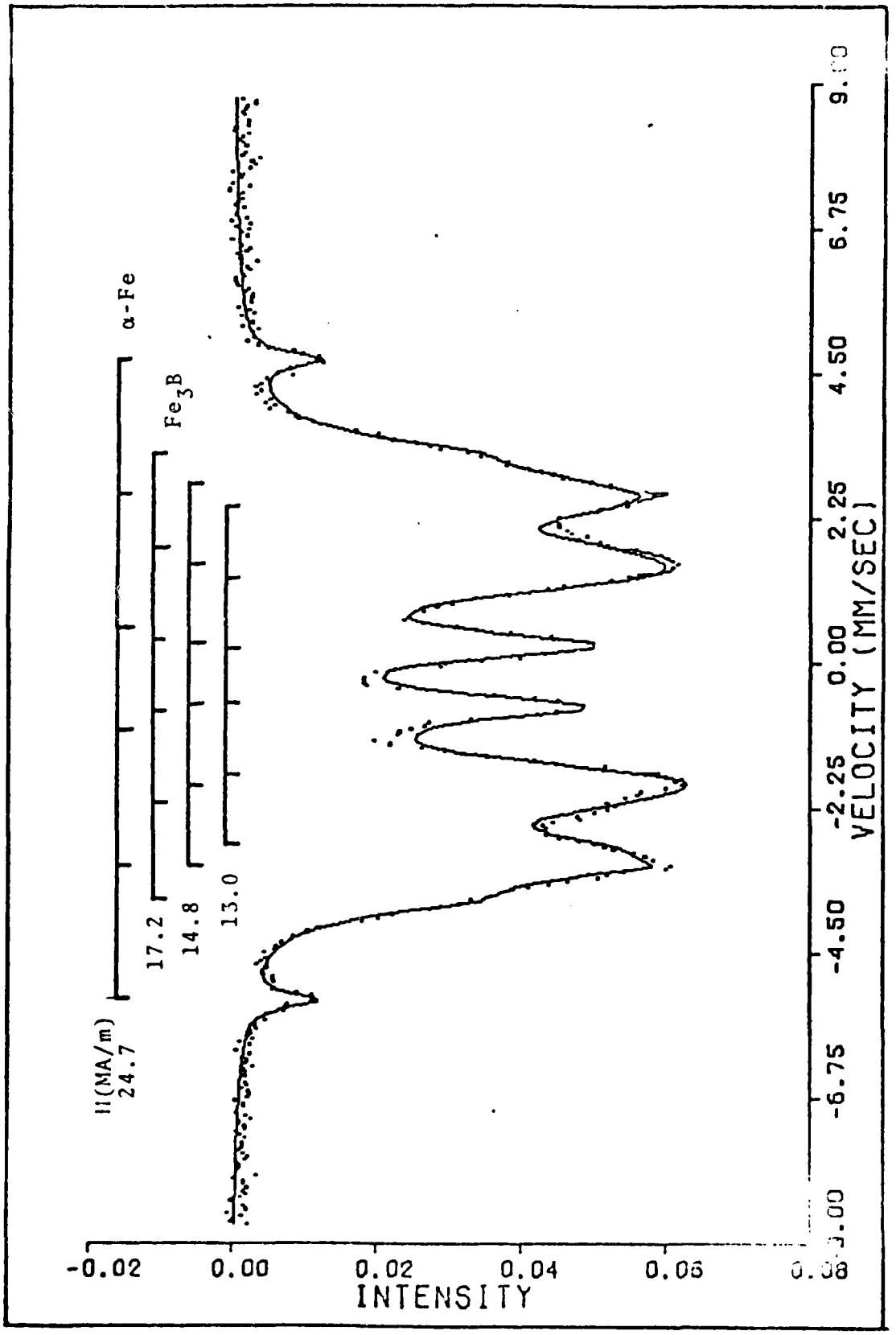

Fig 7. Mossbauer Spectrum of $Fe_{80}B_{20}$, 573 K, 281 Hr, FIVECALF, x = 0.190

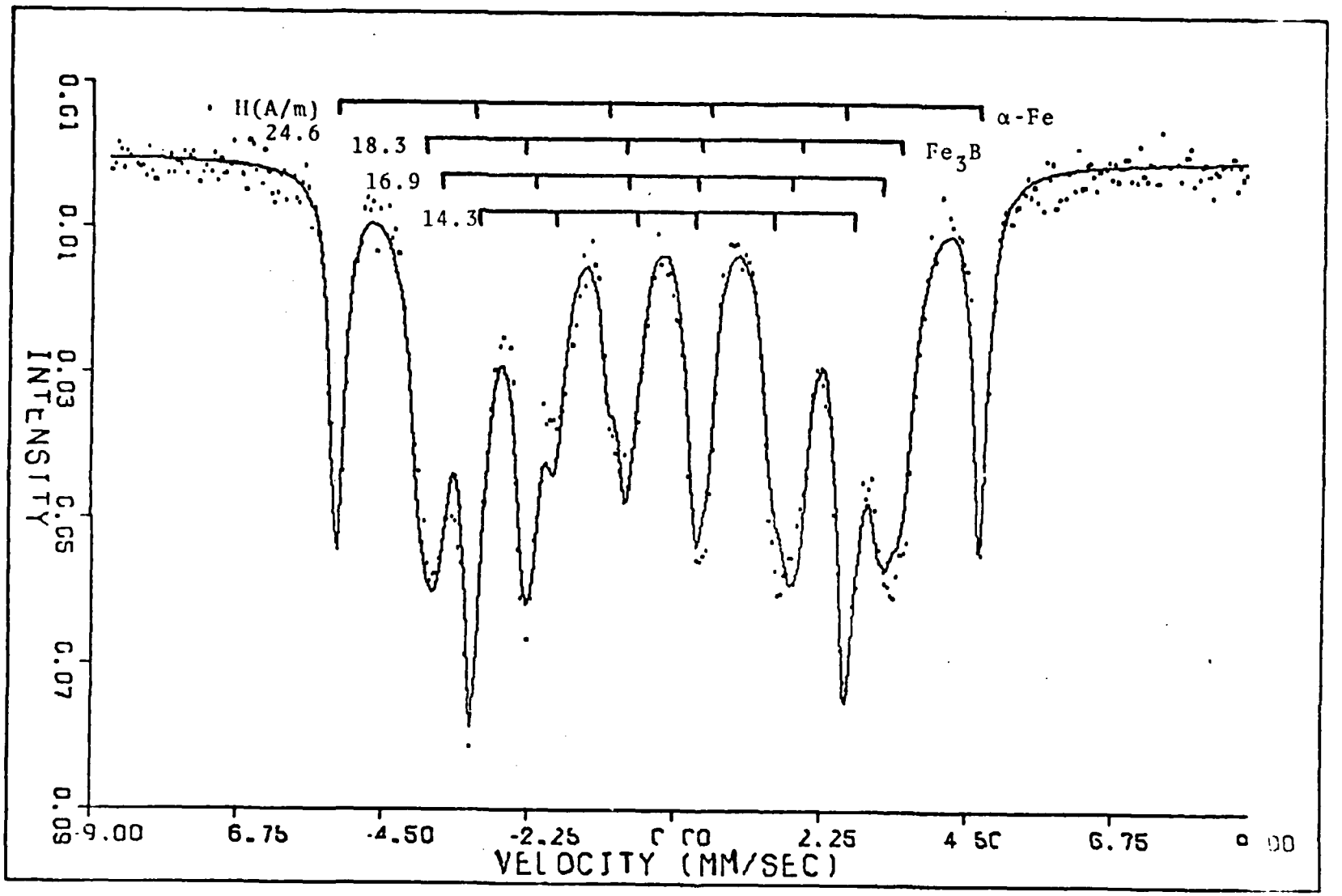

Fig. 8. Mossbauer Spectrum of $Fe_{80}B_{20}$, 573 K, Fully Crystallized, FIVECALF

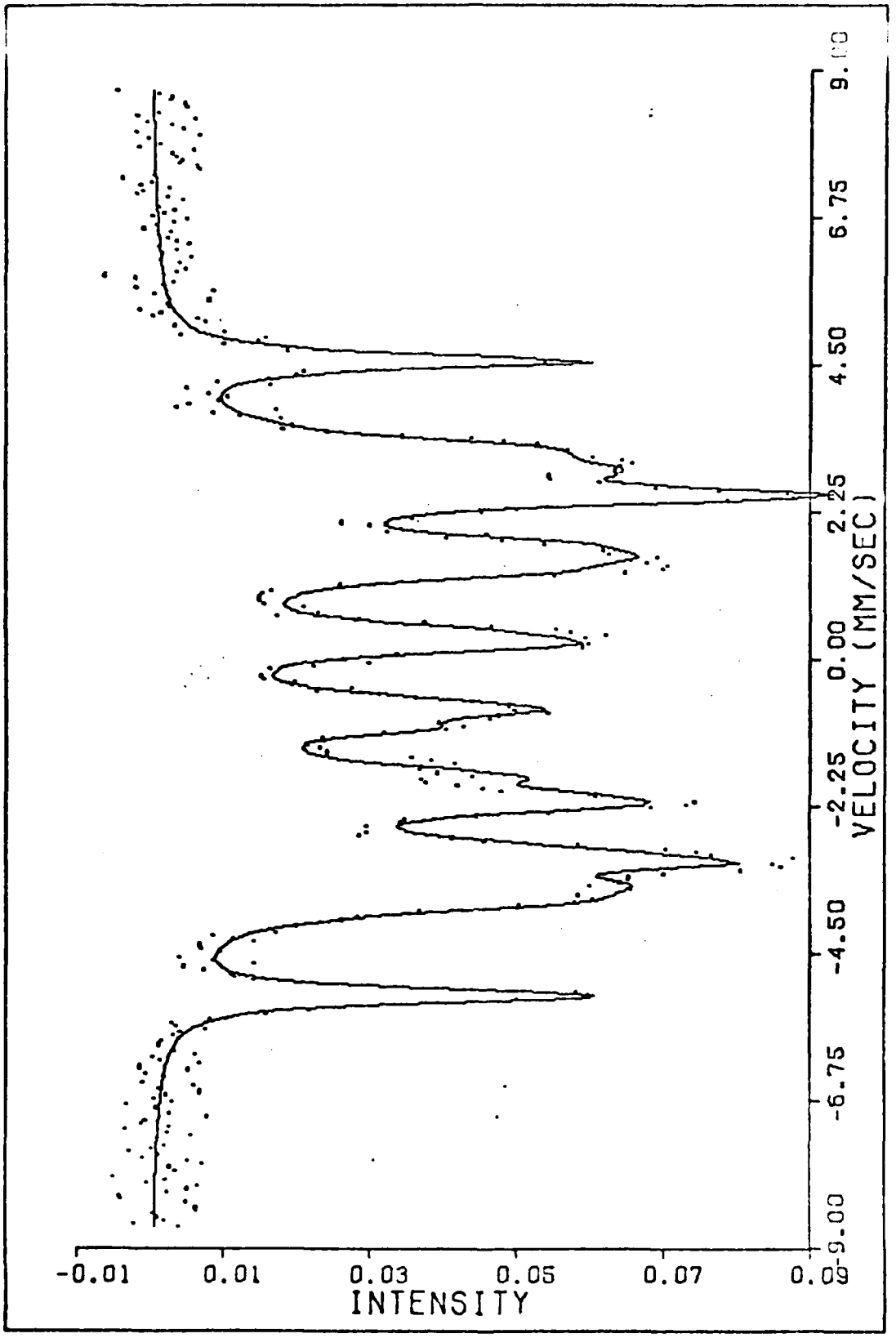

Fig 9. Mossbauer Spectrum of $Fe_{80}B_{20}$, 611 K, Fully Crystallized, FIVECALF

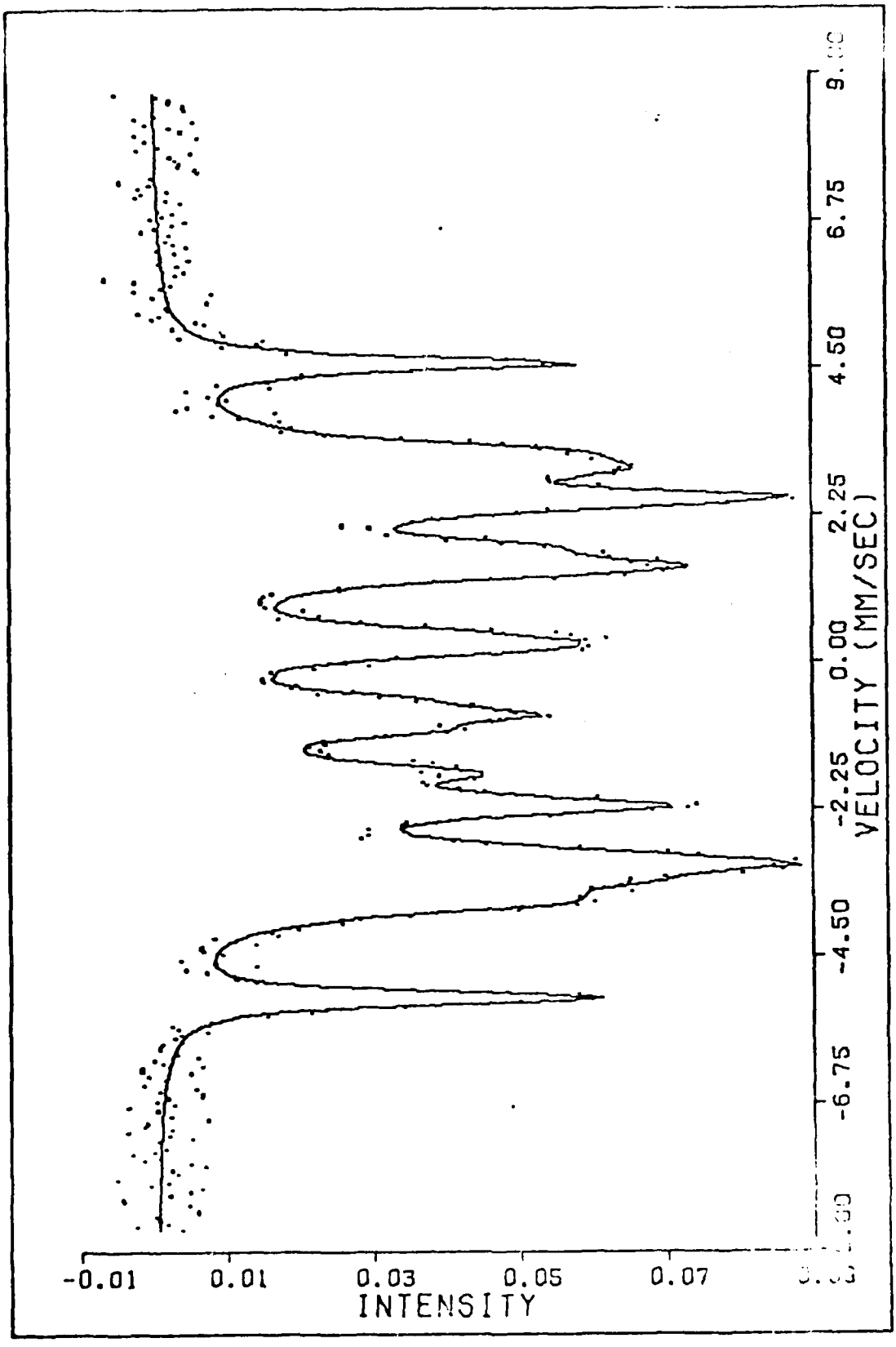

Fig 10. Mossbauer Spectrum of $Fe_{80}B_{20}$, 611 K, Fully Crystallized FIVECALF with Quadrupole Splitting

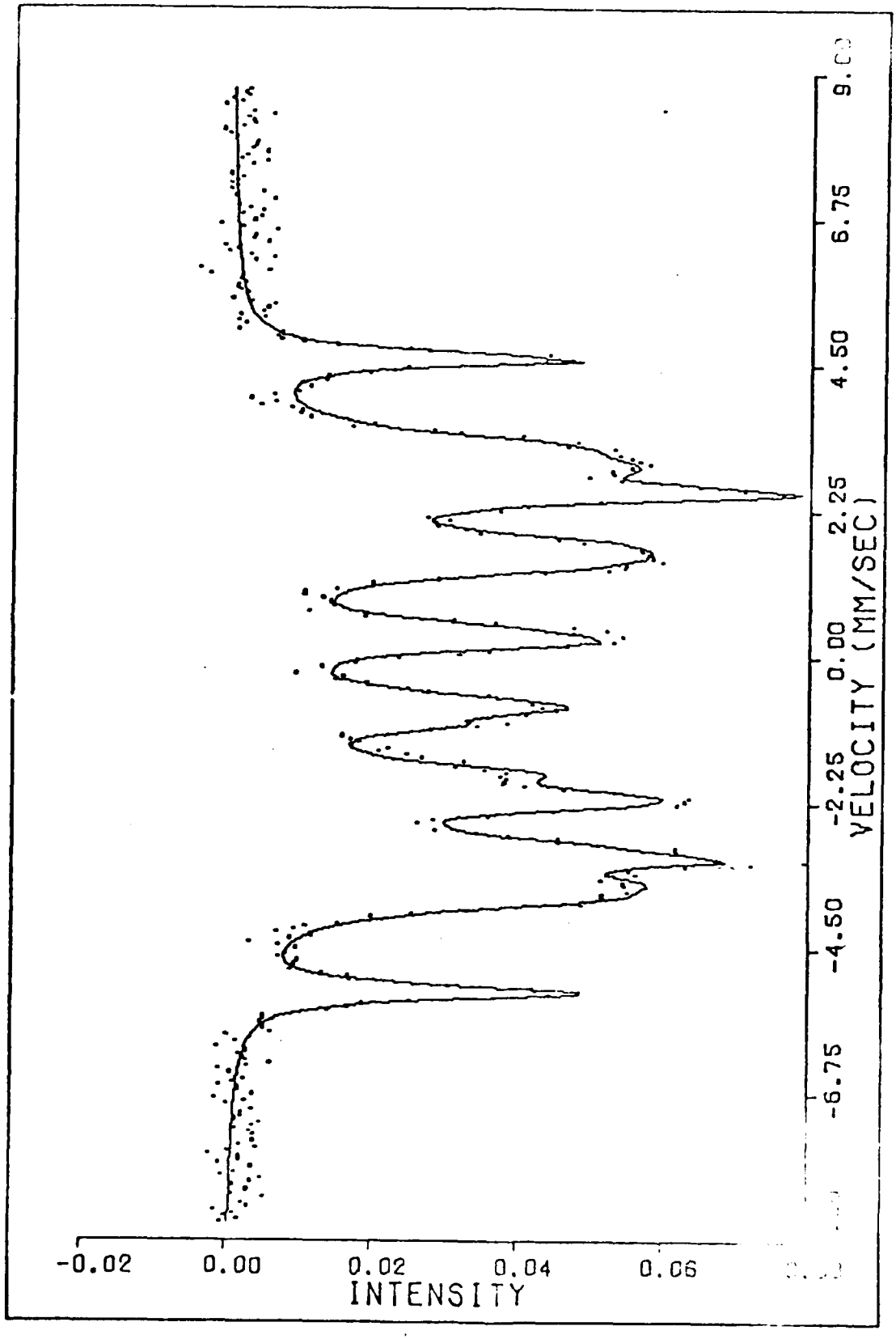

Fig. 11. Mossbauer Spectrum of $Fe_{80}B_{20}$, 611 K, Run 3, Fully Crystallized
FIVECALF

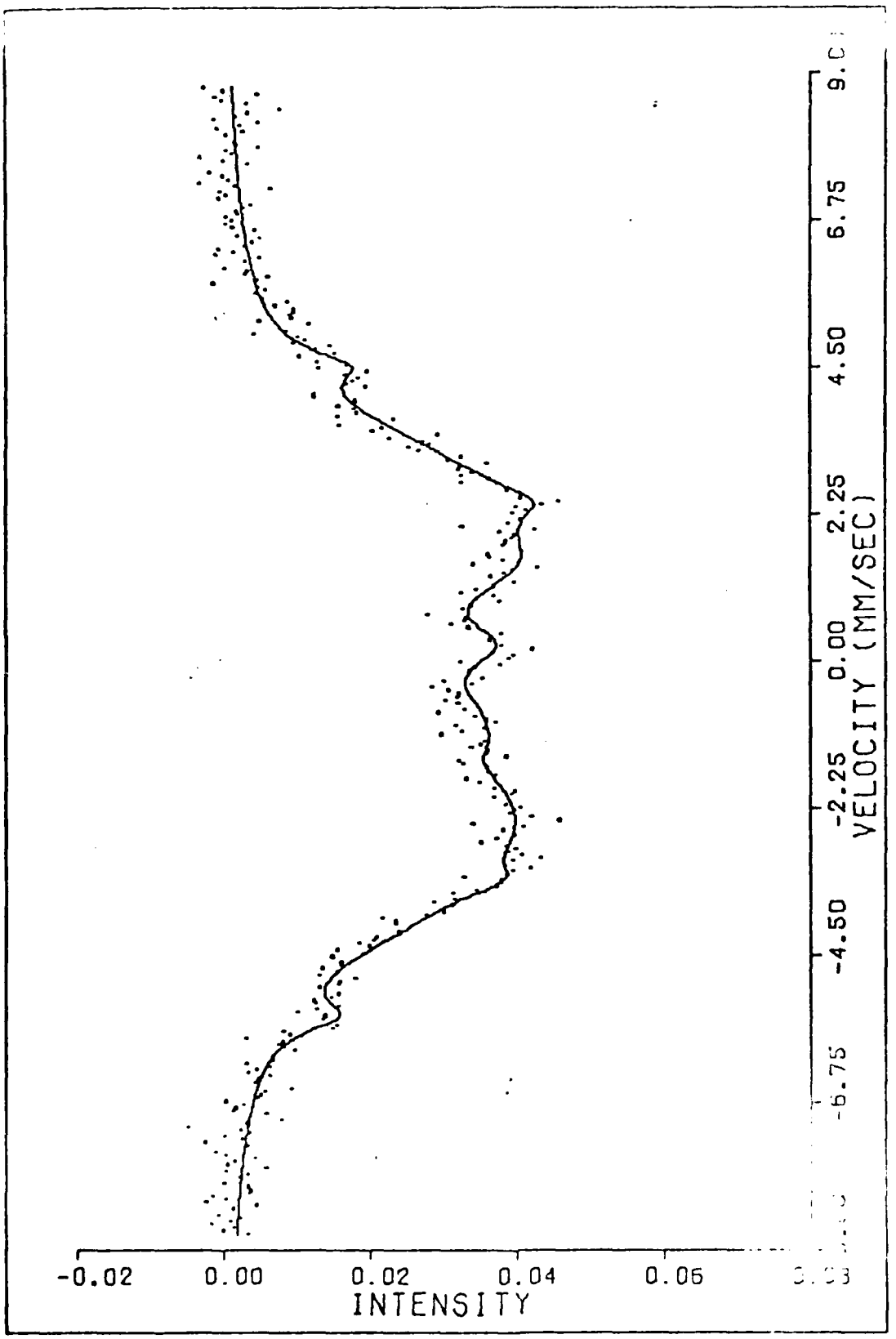

Fig 12. Mossbauer Spectrum of $Fe_{80}B_{20}$, 604 K, 13.8 Hr, FIVECALF, x = 0.316

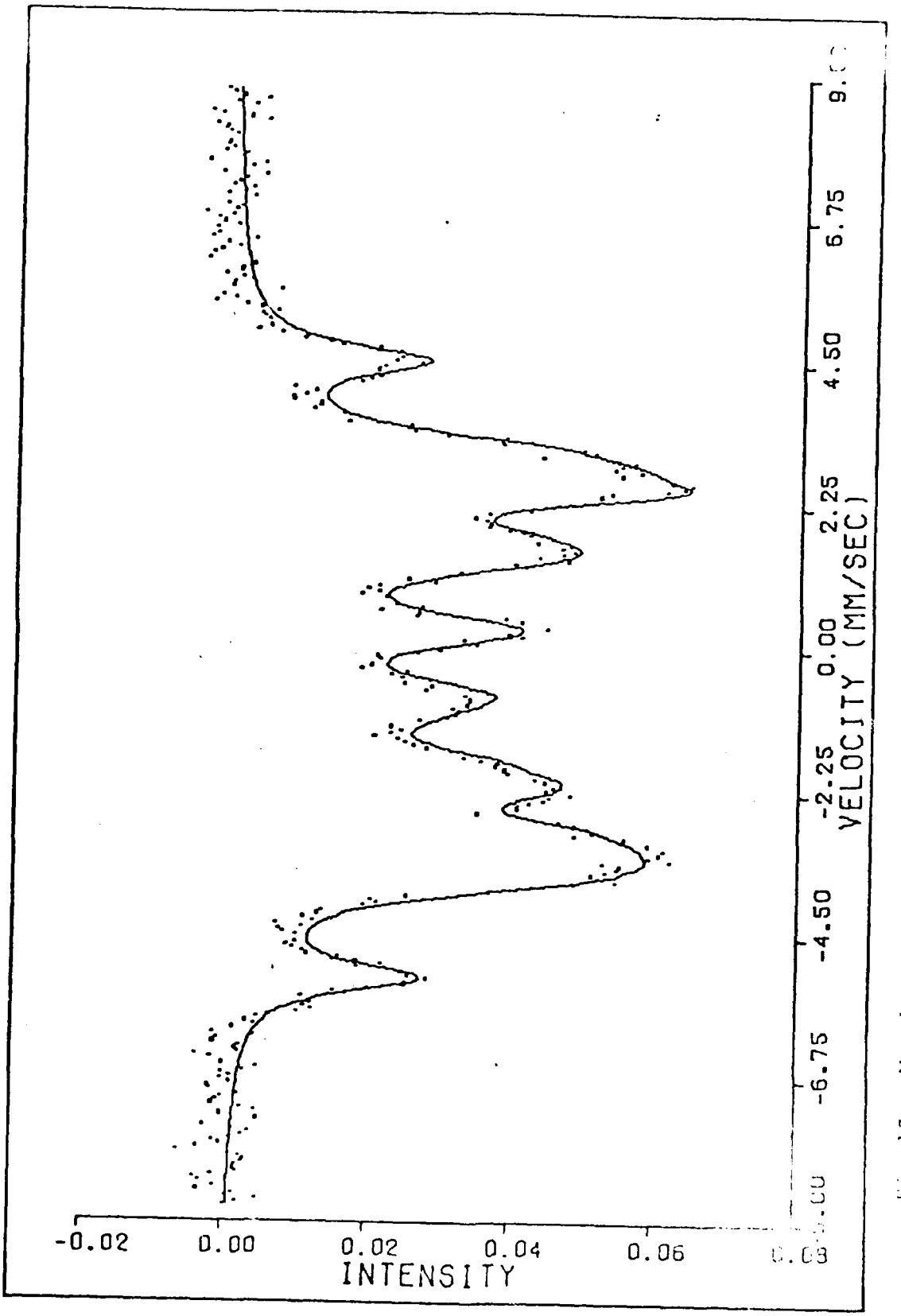

Fig. 13. Mossbauer Spectrum of $Fe_{80}B_{20}$, 604 K, Fully Crystallized, FIVECALF

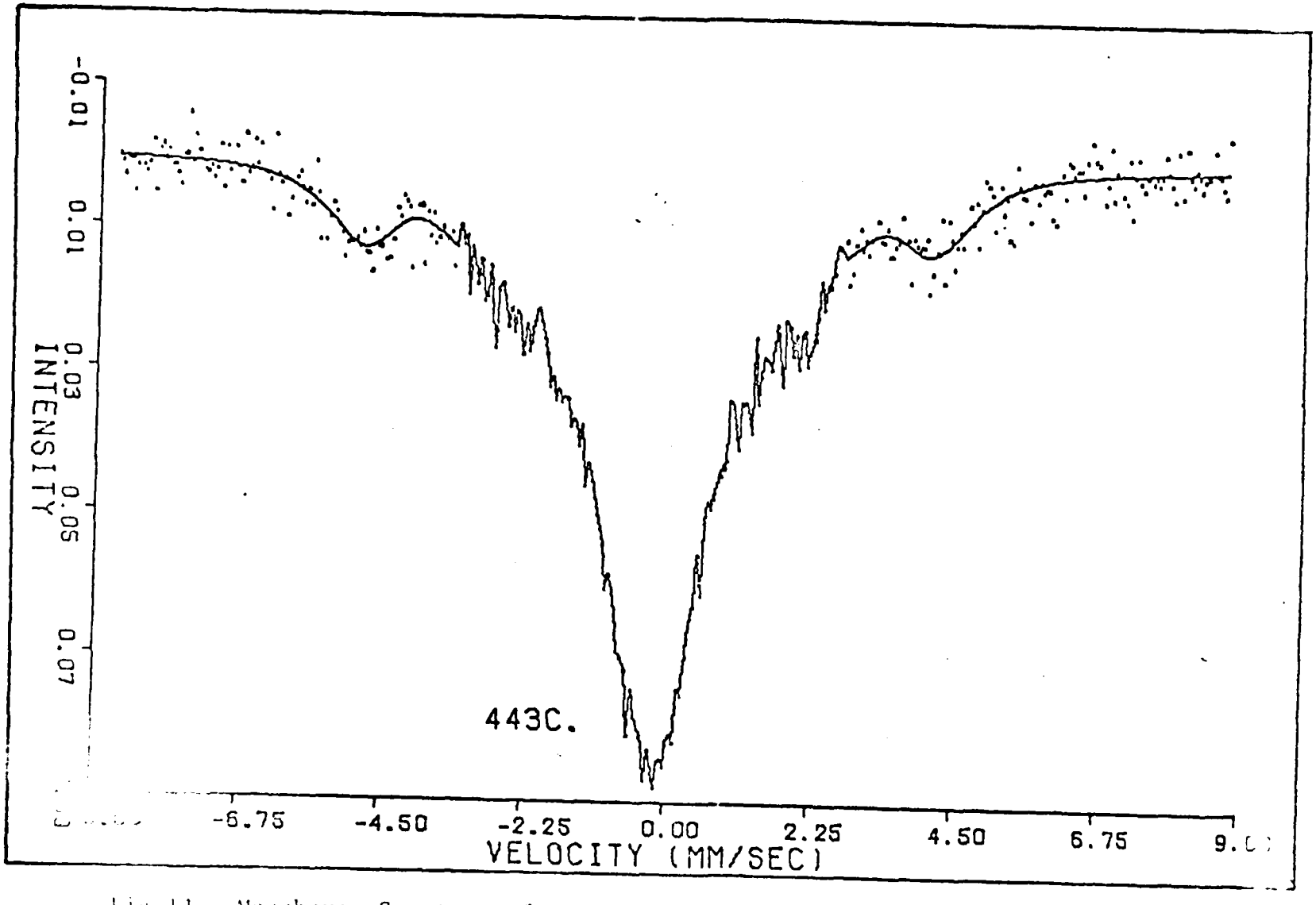

Fig. 11. Mossbauer Spectrum of $Fe_{80}P_{6.5}C_{3.5}B_{10}$, 716 K, 2.87 Hr, ALPHA-BG, x ≅ 1

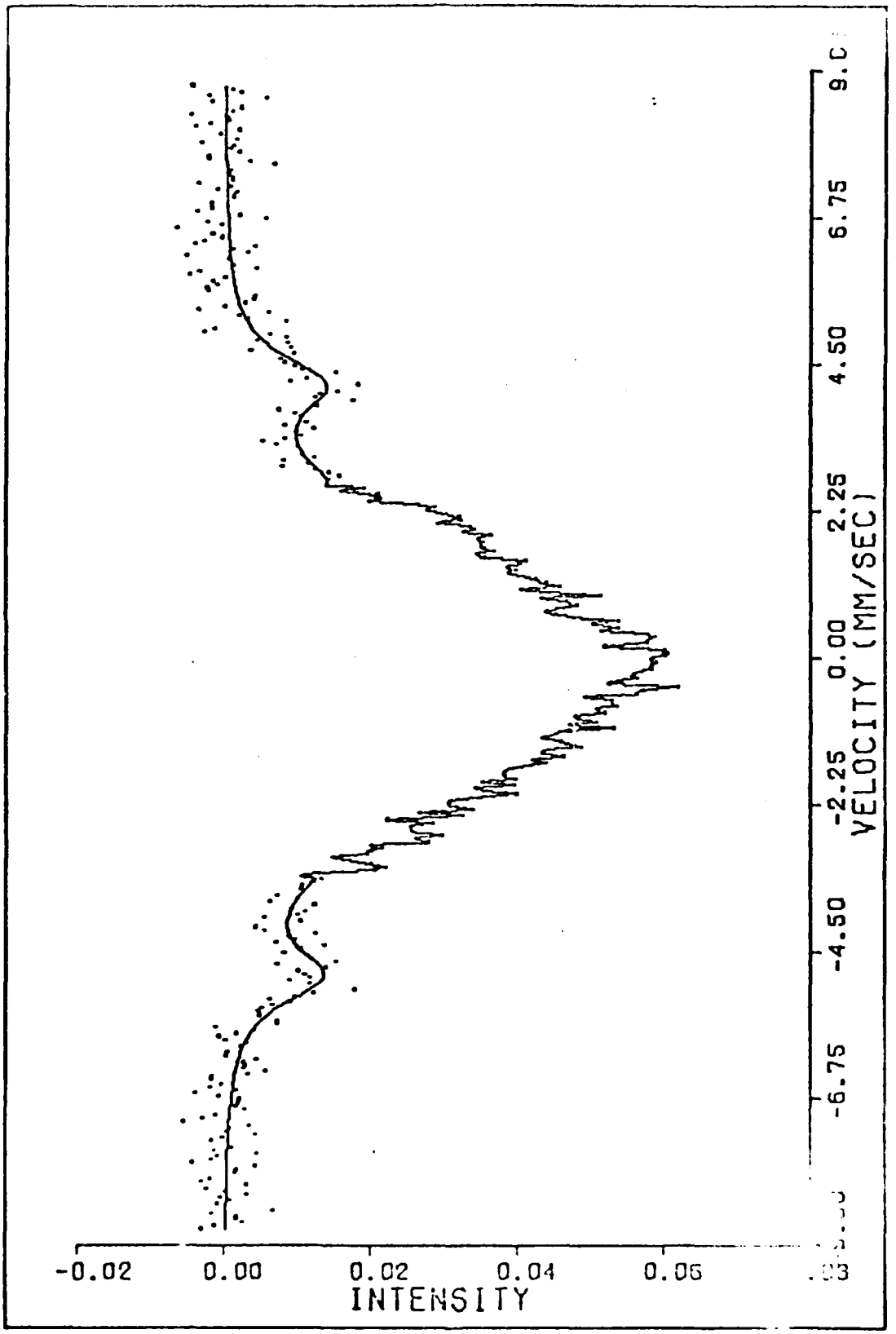

Fig. 15. Mossbauer Spectrum of $Fe_{80}P_{6.5}C_{3.5}B_{10}$, 716 K, Fully Crystallized
ALPHA-BG

clearly demonstrate the effect of constraining the [illegible] quadrupole splits to zero: Chi-squared was 508 for Fig 9, but with quadrupole splitting included in FIVECALF, it was 326 (for 330 data points). Figure 11 is the fully crystallized spectrum of Run 4, and except for total intensity, it is nearly identical to Fig 9 (Run 3 at the same temperature). Figures 12 and 13 show the poorly resolved spectra obtained when using AC power for the heater. Sample motion induced by an alternating magnetic field caused extreme line-broadening which resulted in overlapping of the absorption lines. Figures 14 and 15 are two spectra obtained with $Fe_{80}P_{6.5}C_{3.5}B_{10}$ (Run 7). Although the intensity of the α-Fe peak of Fig 14 indicated a fully crystallized state, visual inspection of these two spectra show a substantial change.

The crystallized fractions for $Fe_{80}B_{20}$ (Runs 1 thru 5) are plotted against time in Fig 16. Representative error bars are indicated on the first and last points of the 626 K data. The data of Run 4 were plotted such that the first data point fell on the calculated Run 3 line. Had the third data point been plotted on this line, the remaining points would have fallen very close to the Run 3 data. Because of low counts and line-broadening of the Mossbauer spectra of Run 2, only the last three data points were useable, and are included in Fig 16. The crystallization half lives, determined from least-squares fits to the data of Fig 16 (between x = 0.[illegible] and x = 0.80). are plotted versus time in Fig 17. The value of the activation energy E_A for α-Fe crystallization was determined

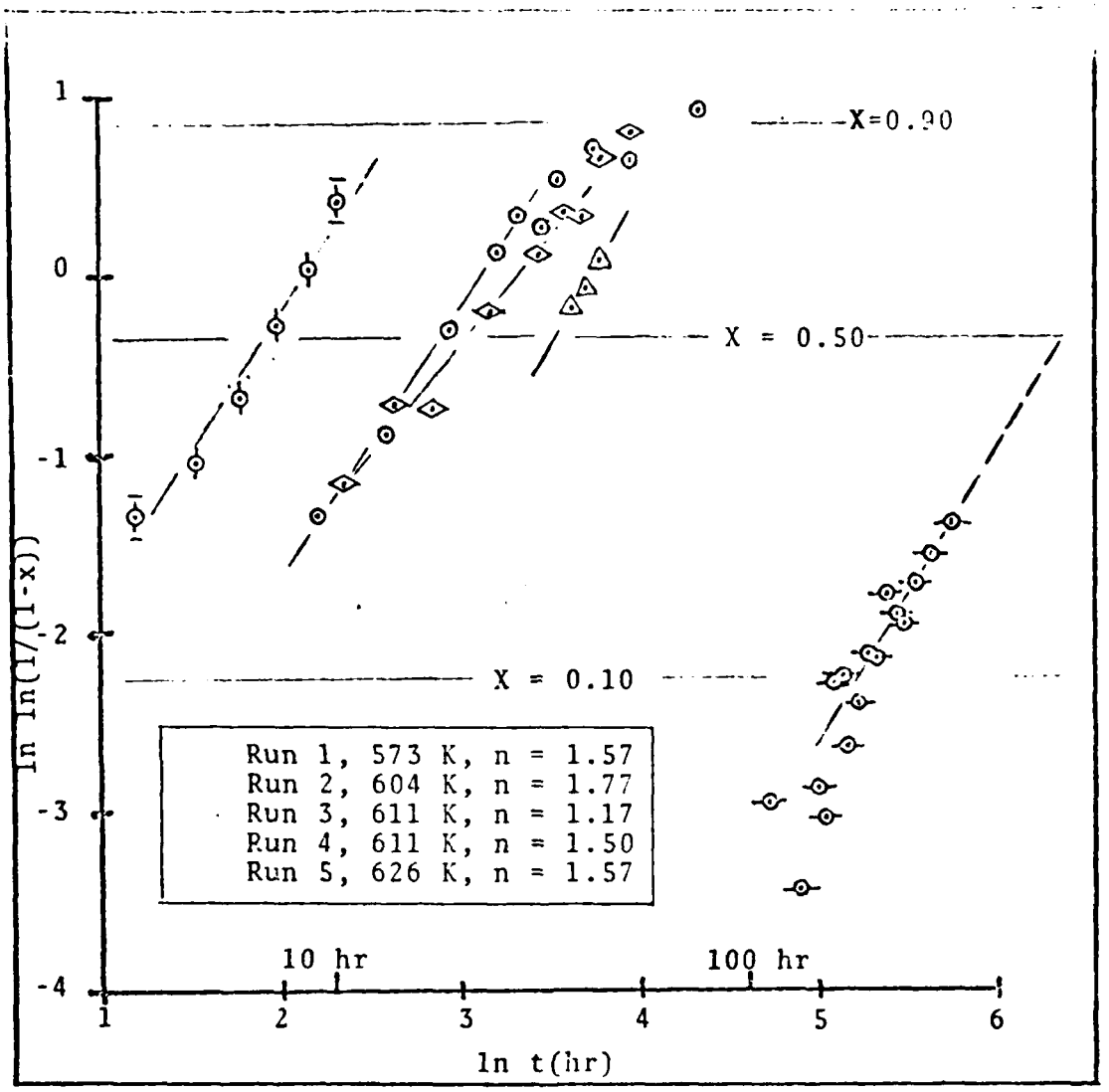

Fig 16. Crystallized Fraction x(t) vs Time, Plotted as ln ln(1/(1-x)) vs ln t

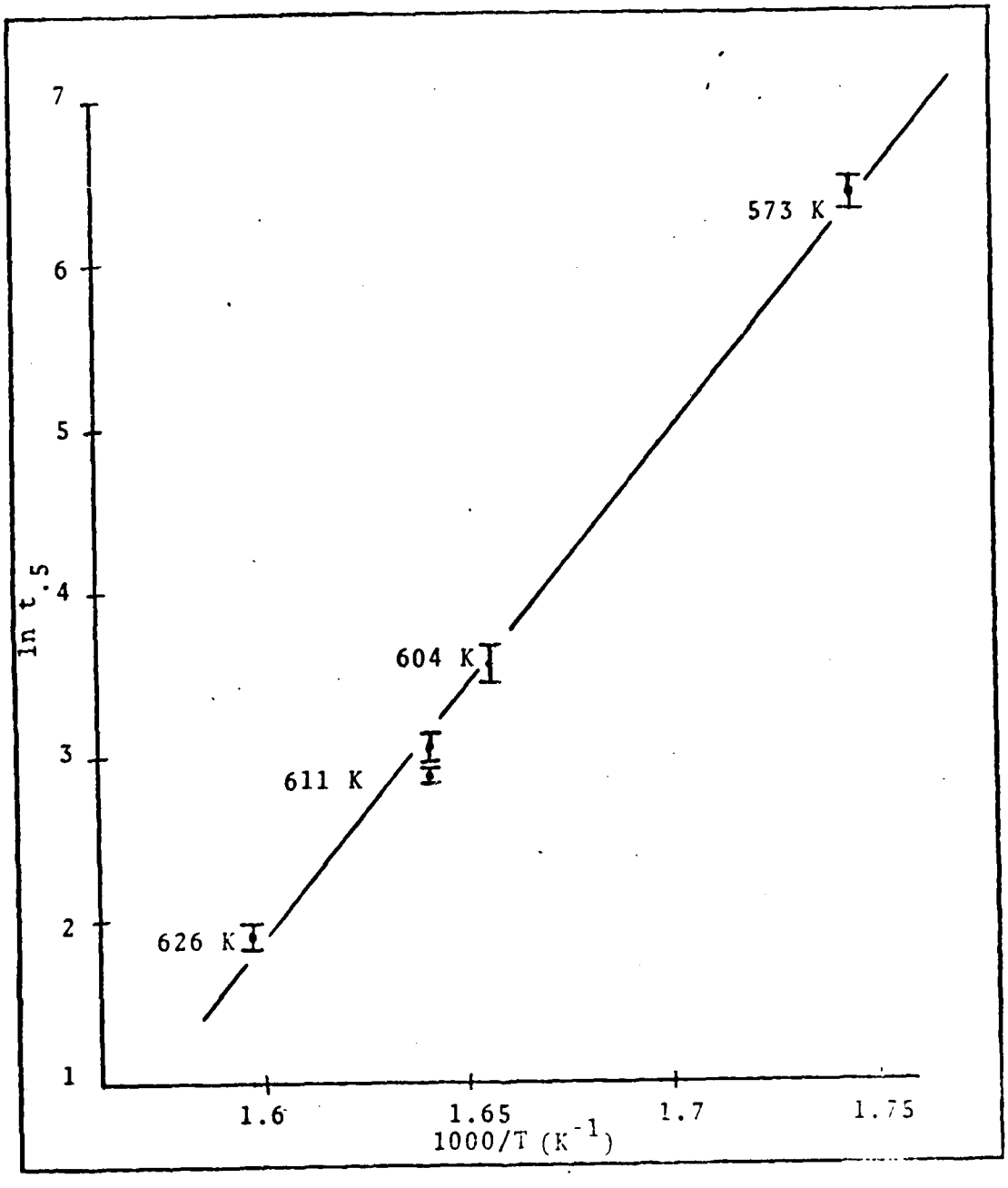

Fig 17. Arrhenius Plot. Crystallization half lives of $Fe_{80}B_{20}$ versus time, plotted as ln $t_{.5}$ vs 1000/T (with 1σ error bars)

from a least-squares fit to the data of this Arrhenius plot. The value of E_A was 0.256 ± 0.006 MJ/mole (2.65 ± 0.07 eV/atom), and ln k_o^{-1} was -47.5. Using these constants and T = 473 K in Eq (3), the predicted half-life of this $Fe_{80}B_{20}$ sample is 5500 ± 450 years at the expected operating temperature of 473 K.

The $Fe_{80}P_{6.5}C_{3.5}B_{10}$ did not crystallize at 614 K, but crystallized extremely fast at 716 and 744 K. Only one or two spectra were taken before full crystallization at these two temperatures. Hence, no kinetic data were obtained for this material. Only qualitative statements can be made about the crystallization of $Fe_{80}P_{6.5}C_{3.5}B_{10}$; these will be presented in the following section.

Discussion

The values of Chi-squared obtained when analyzing the Mossbauer spectra of Runs 1 through 5 varied over a large range--from less than the number of data points for some runs to up to four times the number of data points for others. This indicates a failure of the mathematical model used to describe the Mossbauer spectra. However, this failure was expected, considering the large number of constraints on the model used for FIVECALF (the values of Chi-squared for the GAUSSCALF and ALPHA-BG fits were generally very close to the number of data points). By visual inspection, the fits were good in the area of the α-Fe peaks, and its growth was probably followed accurately. The same cannot be said for the growth

of [illegible], due to the change in the hyperfine field and FWHM during crystallization. Since the glass spectrum does broaden slightly during crystallization, it may continue to change throughout the transformation. The fit to the Fe_3B spectra would then have to account for this change. An alternative view might be to consider the Fe_3B as still semi-amorphous during crystallization.

In Mossbauer spectra taken during early annealing runs, the values of FWHM for the Fe_3B peaks increased for peaks farther from the center. This is typical of glass spectra (Ref 16: 821). For the fully crystallized spectra, the FWHM's were nearly constant, which indicates a crystalline state. This view tends to support Kemeny's (Ref 10: 485) and Schaafsma"s (Ref 11: 4429) conclusions that crystal nuclei exist in the as-quenched glass and that the structure should be based on a locally distorted, quasi-crystalline Fe_3B. It appears that the material is transformed from a glass to a semi-amorphous Fe_3B, and finally to a tetragonal Fe_3B crystal. This may be due to a stress relaxation rather than a crystallization process.

Because of the long time required to reach full crystallization at 573 K, Run 1 was terminated at 315 hrs, and the temperature was raised to 611 K. This was done to try to detect a change in the crystallization process at the lower temperature. The first crystallization time of Run [illegible] was adjusted by Δt so that this data point fell on the line calculated for Run 3. The other times of Run 4 were then adjusted by this

s , and the data was compared to that of Run 3. Because of the data scatter, no conclusion was made concerning a change in the crystallization rate or process.

Using the methods described in Chapter I, others have found the activation energy of $Fe_{80}B_{20}$ to be between 0.195 and 0.257 MJ/mole, with the average toward the upper value (Refs 3: 42; 6: 139; 10: 485; 11: 4427). This range is significant: Luborsky, using calorimetric and magnetic methods to measure the onset of crystallization in $Fe_{80}B_{20}$, determined that the activation energy was 0.202 MJ/mole. He used this value to calculate an expected lifetime of 25 years at 473 K. He determined that "after the onset of crystallization, the magnetic properties deteriorate catastrophically [Ref 3: 139]." Schaafsma determined that the onset of crystallization corresponds to a crystallized fraction of less than 0.02 (Ref 11: 4425). Using his data, with E_A = 0.242 MJ/mole, to calculate the onset of crystallization, the projected lifetime of his glass is 1000 years at 473 K. The same calculation yields a lifetime of 400 years at 473 K for the $Fe_{80}B_{20}$ of this study. These calculations assume that the crystallization rate is determined by the growth of α-Fe, not Fe_3B; and that the crystallization mechanism does not change between 573 and 473 K.

The data obtained during the crystallization of $Fe_{80}P_{6.5}C_{3.5}B_{10}$ were insufficient for analysis of its crystallization kinetics. However, a much higher temperature was required for crystallization. Therefore, qualitatively, its activation energy would be greater than that of $Fe_{80}B_{20}$. This

is true for other similar glasses with many atomic species (i.e., $Fe_{40}Ni_{40}B_{20}$, $Fe_{80}P_{13}C_7$, $Fe_{40}Ni_{40}P_{14}B_6$, Ref 3: 140). Thus, the lifetime of $Fe_{80}P_{6.5}C_{3.5}B_{10}$ is expected to be greater than that of $Fe_{80}B_{20}$.

V. Conclusions and Recommendations

The metallic glasses $Fe_{80}B_{20}$ and $Fe_{80}P_{6.5}C_{3.5}B_{10}$ have been crystallized by isothermal annealing at high temperatures. The crystallization was followed with Mossbauer spectroscopy, and the rates of formation of α-Fe crystals in $Fe_{80}B_{20}$ were determined. These rates were used to determine the activation energy and rate constant of the crystallization process. These constants were used to calculate the expected lifetime of $Fe_{80}B_{20}$ at 473 K, 100 K below the lowest isothermal run. If the crystallization mechanism does not change between 473 and 573 K, the onset of crystallization is projected to be approximately 400 years. No kinetic data were obtained for the crystallization of $Fe_{80}P_{6.5}C_{3.5}B_{10}$, or for the formation of Fe_3B in the $Fe_{80}B_{20}$.

Recommendations

This study can be expanded in the following ways:

1) Use a stronger Mossbauer source to increase counts per channel. This would permit a more careful study of the crystallized spectra to determine Fe_3B growth rates.

2) Use an accurate temperature controller (with direct current), and continue the study at lower temperatures.

3) Study the magnetic and material properties of partially crystallized samples. This would relate crystallized fraction to material performance.

4) Since an alternating field can accelerate the aging of the glass (Ref 12), include a strong rf field during some of the isothermal annealing runs. The field strength must be great enough to displace the atoms in the amorphous metal. Kopcewicz found that 800 A/M at 67 MHz was enough to cause crystallization, but 400 A/M at 53 MHz was not.

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```
      SUBROUTINE CALFUN(NP,NPAR,F,X)                                    000010
C                                                                       000110
C  THIS VERSION OF CALFUN USES A GAUSSIAN LINE SHAPE TO FIT THE         000120
C  ABSORPTION SPECTRUM OF A METALLIC GLASS.  IT GIVES ONE AVERAGE       000130
C  VALUE FOR HYPERFINE FIELD, ONE FOR ISOMER SHIFT, AND ONE FOR         000140
C  QUADRUPOLE SPLIT.  IT GIVES A LINEWIDTH FOR EACH OF THE SIX          000150
C  PEAKS, AND THE INTENSITY RATIO OF PEAK 2 TO PEAK 1. THE AREAL        000160
C  RATIO FOR PEAKS 3 AND 4 TO PEAK 1 IS 1:3.  THESE VALUES CAN          000170
C  THEN BE SUBSTITUTED INTO THE CALFUN CALLED FIVECALF, TO PERMIT       000180
C  FITTING THE CRYSTALLIZED GLASS SPECTRA.                              000190
C                                                                       000200
C              GAUSSCALF   15 OCT 80                                    000210
C                                                                       000220
C     THE REQUIRED VARIABLES ARE:                                       000230
C                                                                       000240
C          ONE VALUE FOR H-FIELD: X(1)                                  000250
C                                                                       000260
C          ONE VALUE OF ISOMER SHIFT: X(2)                              000270
C                                                                       000280
C          ONE VALUE OF QUAD SPLIT: X(3)                                000290
C                                                                       000300
C          ONE VALUE FOR TOTAL INTENSITY OF PEAK 1: X(4)                000310
C                                                                       000320
C          ONE VALUE FOR RELATIVE INTENSITY TO PEAK 1: X(5)             000330
C                                                                       000340
C          SIX VALUES OF LINE WIDTH: X(5) TO X(11)                      000350
C                                                                       000360
C          BASELINE IS LAST VARIABLE: X(12)                             000370
C                                                                       000380
C          TOTAL OF 12 VARIABLES REQUIRED                               000390
C                                                                       000400
C                                                                       000410
      COMMON W(13,31),XD(402),YD(402),YC(402),YDY(402),YCY(402),FSQ(402)000420
      COMMON /HEADING/TITL(18)                                          000430
      COMMON /NAM/XINIT(25),PRM(25),ERX(25),NBASE                       000440
      COMMON /CALF/ IFLAG                                               000450
      DIMENSION F(402),X(25)                                            000460
      REAL V(6),MUEX,MUGND,MUSUBN,LIGHT                                 000470
C                                                                       000480
```

```
C
C  LOAD VALUES OF BINOMIAL PROBABILITIES
C
C
      MUEX=.15491
      MUGND=-.964206
      R=MUEX/(MUGND*3.)
C
C        FACTOR CONVERTS FROM ENERGY UNITS TO VELOCITY UNITS.
C
C
C                              GZERO*MUSUBN*LIGHT*F1*F2
C                    FACTOR = ------------------------
C                                    2.*EZERO*F3
C
      GZERO=.19648
      MUSUBN=5.05E-27
      LIGHT=2.998E11
      F1=1.053
      F2=1.0E-4
      EZERO=14.4125E3
      F3=1.602E-19
C
      FACTOR=(GZERO*MUSUBN*LIGHT*F1*F2)/(EZERO*F3*2.)
C
      A=FACTOR*(3.*R-1.)
      B=FACTOR*(  R-1.)
      C=FACTOR*(  R+1.)
C
      IF(IFLAG)25,25,10
C
C  IF IFLAG IS NOT POSITIVE CALFUN IS BEING CALLED FOR PRINTING ONLY
C
   10 CONTINUE
C
C CALCULATE PEAK VELOCITIES FOR EACH SITE
C
      S=X(2)
      D=X(3)/2.
```

```
      X(1)=ABS(X(1))
      V(1)=A*X(1)+S+Q
      V(2)=B*X(1)+S-Q
      V(3)=-C*X(1)+S-Q
      V(4)=C*X(1)+S-Q
      V(5)=-B*X(1)+S-Q
      V(6)=-A*X(1)+S+Q
      DO 33 I=6,11
33    X(I)=ABS(X(I))
29    CONTINUE
C
C CALCULATE THE SPECTRUM
C
      REL=X(5)*X(7)/X(6)
      IF(REL.GT.1.4) X(5)=1.4*X(6)/X(7)
      DO 31 I=1,NP
      YC(I)=0.0
      DO 35 K=1,6
      IF(K.EQ.1) TI=X(4)
      IF(K.EQ.2) TI=X(4)*X(5)
      IF(K.EQ.3) TI=X(4)*X(6)/(3.*X(8))
      IF(K.EQ.4) TI=X(4)*X(6)/(3.*X(9))
      IF(K.EQ.5) TI=X(4)*X(5)*X(7)/X(10)
      IF(K.EQ.6) TI=X(4)*X(6)/X(11)
      KP5=K+5
      YYY=(XO(I)-V(K))**2/(2.*X(KP5)**.4247*X(KP5)*.4247)
      IF(YYY.GT.100.) YYY=100.
35    YC(I)=YC(I)+(TI/SQRT(6.2832*X(KP5)*X(KP5)*.4247*.4247))*EXP(-YYY)
      YC(I)=(1.0-YC(I))*X(NBASE)
31    F(I)=(YO(I)-YC(I))/SQRT(YO(I))
      RETURN
C
C  ON THE FINAL CALL OF CALFUN PRINT THE PEAK VELOCITIES
C
29    CONTINUE
      PRINT 90
```

```
      PRINT*," "                                                        001240
      PRINT*," "                                                        001250
      PRINT*," THE PEAK VELOCITIES  ARE:"                               001260
      PRINT*," "                                                        001270
      PRINT 100,(V(I),I=1,6)                                            001280
      PRINT*,"  "                                                       001290
      PRINT*,"  "                                                       001300
 99   FORMAT(1H1)                                                       001310
100   FORMAT(5X,7F10.4)                                                 001320
      RETURN                                                            001330
      END                                                               001340
```

```
      SUBROUTINE CALFUN(NP,NPAR,F,X)                                    000100
C                                                                       000110
C  CALFUN USES SUPERPOSITION OF FIVE 5-PEAK SPECTRA: ONE FOR            000120
C  ALPHA-FE, THREE FOR FE3B, AND ONE FOR THE GLASS.  THE VARIABLE       000130
C  PARAMETERS ARE LISTED BELOW.  THE LINEWIDTH FOR ALPHA-FE MUST        000140
C  BE ADDED AT LINE 1731. AREA RATIOS ARE NORMALIZED TO PEAK 1          000150
C  FOR EACH SPECTRUM.  ALL THREE FE3B SPECTRA ARE REQUIRED TO HAVE      000160
C  THE SAME AMPLITUDE, AND AREA RATIOS FOR PEAKS 1:2:3:4:5:6 OF         000170
C  3:2:1:1:2:3.  FOR THE GLASS SPECTRUM, ONLY THE INTENSITY IS A        000180
C  VARIABLE PARAMETER, THE OTHERS MUST BE SUPPLIED IN LINES 1461        000190
C  TO 1481 AND 1821 TO 1920.  THIS CALFUN ALSO GIVES THE COUNTS         000200
C  ABSORBED BY ALPHA-FE AND THE TOTAL COUNTS ABSORBED. THIS VERSION     000210
C  OF FIVECALF IS FOR 336 C DATA ONLY.                                  000220
C                                                                       000230
C                                                                       000240
C               FIVECALF    22 NOV 80                                   000250
C                                                                       000260
C    THE REQUIRED VARIABLES ARE:                                        000270
C                                                                       000280
C         ONE VALUE OF H-FIELD FOR ALPHA IRON: X(1)                     000290
C                                                                       000300
C         THREE VALUES OF H-FIELD FOR FE3B: X(2) TO X(4)                000310
C                                                                       000320
C         ONE VALUE OF TOTAL INTENSITY FOR ALPHA FE: X(5)               000330
C                                                                       000340
C         ONE VALUE OF TOTAL INTENSITY FOR FE3B: X(6)                   000350
C                                                                       000360
C         THREE VALUES OF LINEWIDTH FOR FE3B: X(7) TO X(9)              000370
C                                                                       000380
C         ONE VALUE OF ISOMER SHIFT FOR ALPHA FE: X(10)                 000390
C                                                                       000400
C         THREE VALUES OF ISOMER SHIFT FOR FE3B: X(11) TO X(13)         000410
C                                                                       000420
C         ONE VALUE OF TOTAL INTENSITY FOR THE FE*B20:  X(14)           000430
C                                                                       000440
C         BASELINE IS LAST VARIABLE: X(15)                              000450
C                                                                       000460
C         TOTAL OF 15 VARIABLES REQUIRED                                000470
C                                                                       000480
```

```
C                                                                       000100
      COMMON W(13(3[),XC(402),YD(402),F3(402),YDY(402),YCY(402),FSO(402)000110
      COMMON /HEADING/TITL(18)                                          000120
      COMMON /NAM/XINIT(25),PRM(25),ERX(25),NBASE                       000130
      COMMON /CALF/ IFLAG                                               000140
      DIMENSION F(402),X(25)                                            000150
      REAL  V(30),MUEX,MUGND,MUSUBN,LIGHT,H,B1(5)                       000160
C                                                                       000170
C                                                                       000180
C                                                                       000190
C                                                                       000200
      MUEX=.11491                                                       000210
      MUGND=-.19[12[E                                                   000220
      R=MUEX/(MUGND*3.)                                                 000230
C                                                                       000240
C        FACTOR CONVERTS FROM ENERGY UNITS TO VELOCITY UNITS.           000250
C                                                                       000260
C                                                                       000270
C                                GZERO*MUSUBN*LIGHT*F1*F2               000280
C                     FACTOR = ------------------------                 000290
C                                    2.*EZERO*F3                        000300
C                                                                       000310
      GZERO=.18048                                                      000320
      MUSUBN=5.05E-27                                                   000330
      LIGHT=2.998E11                                                    000340
      F1=1.6E3                                                          000350
      F2=1.0E-4                                                         000360
      EZERO=10.4125E3                                                   000370
      F3=1.602E-19                                                      000380
C                                                                       000390
      FACTOR=(GZERO*MUSUBN*LIGHT*F1*F2)/(EZERO*F3*2.)                   000400
C                                                                       000410
      [ = FACTOR*(3.*R-1.)                                              000420
      [ = FACTOR*(  R-1.)                                               000430
      [ = FACTOR*(  R+1.)                                               000440
C                                                                       000450
      IF(IFLAG)25,25,10                                                 000460
C                                                                       000470
C IF IFLAG IS NOT POSITIVE CALFUN IS BEING CALLED FOR PRINTING ONLY     000480
```

```
C                                                                       001180
10    CONTINUE                                                          001190
C                                                                       001200
C  CALCULATE PEAK VELOCITIES FOR EACH SITE                              001210
C                                                                       001220
      DO 11 I=1,9                                                       001230
11    X(I)=ABS(X(I))                                                    001240
C                                                                       001250
C  THE FOLLOWING IS FOR THE ALPHA IRON SPECTRUM.                        001260
C  H IS THE HYPERFINE FIELD AND S IS THE ISOMER SHIFT.                  001270
C  V(1) TO V(6) ARE THE VELOCITIES OF PEAKS ONE TO SIX.                 001280
C                                                                       001290
      H=X(1)                                                            001300
      S=X(10)                                                           001310
      V(1)=A*H+S                                                        001320
      V(2)=B*H+S                                                        001330
      V(3)=-C*H+S                                                       001340
      V(4)=C*H+S                                                        001350
      V(5)=-B*H+S                                                       001360
      V(6)=-A*H+S                                                       001370
C                                                                       001380
C  THE FOLLOWING IS FOR THE FIRST FE3B SPECTRUM.                        001390
C                                                                       001400
      H=X(2)                                                            001410
      S=X(11)                                                           001420
      Q=0.                                                              001430
      V(7)=A*H+S+Q                                                      001440
      V(13)=B*H+S-Q                                                     001450
      V(19)=-C*H+S-Q                                                    001460
      V(20)=C*H+S-Q                                                     001470
      V(14)=-B*H+S-Q                                                    001480
      V(8)=-A*H+S+Q                                                     001490
C                                                                       001500
C  THE FOLLOWING IS FOR THE SECOND FE3B SPECTRUM.                       001510
C                                                                       001520
      H=X(3)                                                            001530
```

```
      S=X(12)                                                          00 1240
      V(9)=A*H+S+Q                                                     00 1250
      V(15)=D*H+S-Q                                                    00 1260
      V(21)=-C*H+S-Q                                                   00 1270
      V(22)=C*H+S-Q                                                    00 1280
      V(16)=-D*H+S-Q                                                   00 1290
      V(10)=-A*H+S+Q                                                   00 1300
C                                                                      00 1310
C  THE FOLLOWING IS THE THIRD FE3B SPECTRUM.                           00 1320
      H=X( )                                                           00 1330
      S=X(13)                                                          00 1340
      V(11)=A*H+S+Q                                                    00 1350
      V(17)=D*H+S-Q                                                    00 1360
      V(23)=-C*H+S-Q                                                   00 1370
      V(24)=C*H+S-Q                                                    00 1380
      V(18)=-D*H+S-Q                                                   00 1390
      V(12)=-A*H+S+Q                                                   00 1400
C                                                                      00 1410
C  THE FOLLOWING IS THE FE80B20 GLASS SPECTRUM, IT MUST BE CHANGED     00 1420
C  TO MATCH THE NON-CRYSTALLIZED GLASS SPECTRUM FOR A GIVEN            00 1430
C  TEMPERATURE.                                                        00 1440
C                                                                      00 1450
      H=145.                                                           00 1460
      S=-.23                                                           00 1470
      Q=-.05                                                           00 1480
      V(25)=A*H+S+Q                                                    00 1490
      V(35)=D*H+S-Q                                                    00 1500
      V(27)=-C*H+S-Q                                                   00 1510
      V(28)=C*H+S-Q                                                    00 1520
      V(29)=-D*H+S-Q                                                   00 1530
      V(31)=-A*H+S+Q                                                   00 1540
20    CONTINUE                                                         00 1550
C                                                                      00 1560
C CALCULATE THE SPECTRUM                                               00 1570
C                                                                      00 1580
      AAEE=0.0                                                         00 1590
      ATOT=0.0                                                         00 1600
      DO 30 I=1,NP                                                     00 1610
      IF(I.LE.150 .OR. I.GE.151) GO TO 31                              00 1620
```

```
      YC(I)=1-(.05*YD(I-1)+.005*YD(I+1)+.99*YD(I))/X(NBASE)
      GO TO 36
31    YC(I)=1.
      DO 35 K=1,30
      IF(K.EQ.1 .OR. K.EQ.6) TI=X(5)
      IF(K.EQ.2 .OR. K.EQ.5)  TI=X(5)*2./3.
      IF(K.EQ.3 .OR. K.EQ.4) TI=X(5)/3.
      IF(K.GT.6 .AND. K.LT.13) TI=X(5)
      IF(K.GT.12 .AND. K.LT.19) TI=X(5)*X(7)*2./3./X(8)
      IF(K.GT.18) TI=X(6)*X(7)/3./X(9)
      IF(K.LT.7) B=.56
      IF(K.GT.6 .AND. K.LT.13) B=X(7)
      IF(K.GT.12 .AND. K.LT.19) B=X(8)
      IF(K.GT.18 .AND. K.LT.25) B=X(9)
      IF(K.GT.24) GO TO 32
      YCP=TI/((((XD(I)-V(K))**2*4.)/B**2)+1.)
      GO TO 33
32    TI1=ABS(X(14))
C
C
C  THE FOLLOWING ARE THE LINEWIDTHS OF THE SIX GLASS PEAKS.
C
      B1(1)=2.45
      B1(2)=1.640
      B1(3)=1.38
      B1(4)=1.25
      B1(5)=1.76
      B1(6)=2.59
      IF(K.EQ.25) TI=TI1
C
C  THE 1.3 IN THE FIRST AND FOURTH LINES FOLLOWING IS THE
C  INTENSITY RATIO OF PEAK 2 TO PEAK 1 (OF THE GLASS).
C
      IF(K.EQ.26) TI=TI1*1.3*B1(1)/B1(2)
      IF(K.EQ.27) TI=TI1*B1(1)/3./B1(3)
      IF(K.EQ.28) TI=TI1*B1(1)/3./B1(4)
      IF(K.EQ.29) TI=TI1*1.3*B1(1)/B1(5)
      IF(K.EQ.30) TI=TI1*B1(1)/B1(6)
      YYY=(XD(I)-V(K))**2/.3607/B1(K-24)**2
      IF(YYY.GT.100.) YYY=100.
```

```
      YCP=(TI/SQRT(1.133+B1(K-24)**2))*EXP(-YYY)                        00 2520
C                                                                       00 2530
C  CALCULATE COUNTS ABSORBED BY ALPHA-FE.                               00 2540
C                                                                       00 2550
33    IF(K.LT.7) AAFE=AAFE+YCP                                          00 2560
35    YC(I)=YC(I)+YCP                                                   00 2570
C                                                                       00 2580
C  CALCULATE TOTAL COUNTS ABSORBED.                                     00 2590
C                                                                       00 2600
36    ATOT=ATOT+YC(I)                                                   00 2610
      YC(I)=(1.-YC(I))*X(NBASE)                                         00 2620
38    F(I)=(YO(I)-YC(I))/SQRT(YO(I))                                    00 2630
      RETURN                                                            00 2640
C                                                                       00 2650
C  ON THE FINAL CALL OF CALFUN PRINT THE PEAK VELOCITIES AND            00 2660
C  THE COUNTS ABSORBED.                                                 00 2670
C                                                                       00 2680
25    CONTINUE                                                          00 2690
      PRINT 92                                                          00 2700
92    FORMAT(1H1,//,38H  THE PEAK VELOCITIES ARE (IN MM/SEC):,//)       00 2710
      PRINT 1[ ,(V(I),I=1,6)                                            00 2720
      PRINT 93                                                          00 2730
93    FORMAT(//,53H  THE PEAK VELOCITIES FOR THE THREE FE57 SPECTRA ARE: 00 2740
     1,//)                                                              00 2750
      DO 85 I=7,11,2                                                    00 2760
      PRINT 1[ ,V(I),V(I+6),V(I+12),V(I+13),V(I+7),V(I+1)               00 2770
85    PRINT 9[                                                          00 2780
      PRINT 94,AAFE                                                     00 2790
94    FORMAT(/////,43H  THE TOTAL ALPHA IRON AREA (SIX PEAKS) IS://,    00 2800
     1F1[.4,//)                                                         00 2810
      PRINT 95,ATOT                                                     00 2820
95    FORMAT(//,37H  THE AREA UNDER THE ENTIRE CURVE IS:,//,F1[.4)      00 2830
      PRINT 96                                                          00 2840
96    FORMAT(1H1)                                                       00 2850
98    FORMAT(/)                                                         00 2860
1[[   FORMAT(5X,7F1[.4)                                                 00 2870
      RETURN                                                            00 2880
      END                                                               00 2890
```

```
      SUBROUTINE CALFUN(NP,NPAR,F,X)
C
C  THIS CALFUN FITS ONLY PEAKS ONE AND SIX OF THE ALPHA-FE SPECTRUM.
C  IT ADDS A GAUSSIAN SHAPED BACKGROUND TO THE ALPHA-FE TO ACCOUNT FOR
C  THE CLOSENESS OF THE GLASS SPECTRUM TO THE CRYSTALLIZED ALPHA-FE
C  SPECTRUM.  IT FITS THE CENTER PORTION OF THE SPECTRUM WITH THE
C  APPROXIMATE DATA VALUES.
C
C            ALPHA-BG  22 OCT 80
C
C     THE REQUIRED VARIABLES ARE:
C
C          ONE VALUE OF H-FIELD FOR ALPHA IRON: X(1)
C
C          ONE VALUE OF H-FIELD FOR THE BACKGROUND CURVE: X(2)
C
C          ONE VALUE OF ISOMER SHIFT FOR ALPHA IRON: X(3)
C
C          ONE VALUE OF ISOMER SHIFT FOR THE BACKGROUND CURVE: X(4)
C
C          TWO VALUES OF LINE WIDTH: X(5) TO X(5)
C
C          ONE VALUE OF TOTAL INTENSITY OF PEAK 1: X(7)
C            FOR ALPHA IRON
C
C          ONE VALUE OF TOTAL INTENSITY OF THE BACKGROUND: X(8)
C
C          BASELINE IS LAST VARIABLE: X(9)
C
C          TOTAL OF 9 VARIABLES REQUIRED
C
C
      COMMON W(13000),XD(402),YD(402),YC(402),YDY(402),YCY(402),FSD(402)
      COMMON /HEADING/TITL(18)
      COMMON /NAM/XINIT(25),PRM(25),ERX(25),NBASE
```

```
      COMMON /CALF/ IFLAG                                                   000650
      DIMENSION F(402),X(25)                                                000660
      REAL  V(6),MUEX,MUGND,MUSUBN,LIGHT                                    000670
C                                                                           000680
C                                                                           000690
C  LOAD VALUES OF BINOMIAL PROBABILITIES                                    000700
C                                                                           000710
C                                                                           000720
      MUEX=.15491                                                           000730
      MUGND=-.09.4216                                                       000740
      R=MUEX/(MUGND*3.)                                                     000750
C                                                                           000760
C        FACTOR CONVERTS FROM ENERGY UNITS TO VELOCITY UNITS.               000770
C                                                                           000780
C                                                                           000790
C                                  GZERO*MUSUBN*LIGHT*F1*F2                 000800
C                       FACTOR = ------------------------                   000810
C                                      2.*EZERO*F3                          000820
C                                                                           000830
      GZERO=.18648                                                          000840
      MUSUBN=5.05E-27                                                       000850
      LIGHT=2.998E11                                                        000860
      F1=1.0E3                                                              000870
      F2=1.0E-4                                                             000880
      EZERO=14.4125E3                                                       000890
      F3=1.6021E-19                                                         000900
C                                                                           000910
      FACTOR=(GZERO*MUSUBN*LIGHT*F1*F2)/(EZERO*F3*2.)                       000920
C                                                                           000930
      A= FACTOR*(3.*R-1.)                                                   000940
      B = FACTOR*(  R-1.)                                                   000950
      C = FACTOR*(  R+1.)                                                   000960
C                                                                           000970
      IF(IFLAG)25,25,10                                                     000980
C                                                                           000990
C IF IFLAG IS NOT POSITIVE CALFUN IS BEING CALLED FOR PRINTING ONLY         001000
C                                                                           001010
10    CONTINUE                                                              001020
C                                                                           001030
```

```
C CALCULATE PEAK VELOCITIES FOR EACH SITE
C
      DO 11 I=1,2
11    X(I)=ABS(X(I))
      V(1)=A*X(1)+X(3)
      V(2)=A*X(2)+X(4)
      V(3)=-A*X(2)+X(4)
      V(4)=-A*X(1)+X(3)
      DO 33 I=5,8
33     X(I)=ABS(X(I))
20    CONTINUE
C
C CALCULATE THE SPECTRUM
C
      AAFE=0.0
      ATOT=0.0
      DO 3' I=1,NP
      IF(I.LT.192 .OR. I.GT.214) GO TO 31
      YC(I)=1-(.05*YD(I-1)+.05*YD(I+1)+.39*YD(I))/X(NBASE)
      GO TO 36
31    YC(I)=0.
      DO 35 K=1,4
      IF(K.EQ.1 .OR. K.EQ.4) TI=X(7)
      IF(K.EQ.2 .OR. K.EQ.3) TI=X(8)
      IF(K.EQ.2 .OR. K.EQ.3.) GO TO 32
      YCP=TI/((((XD(I)-V(K))**2*4.)/X(5)**2)+1.)
      AAFE=AAFE+YCP
      GO TO 35
32    YYY=(XD(1)-V(K))**2/(2.*(.4247*X(5))**2)
      IF(YYY.GT.100) YYY=100.
      YCP=TI/SQRT(6.2836)/.4247/X(5)*EXP(-YYY)
35    YC(I)=YC(I)+YCP
36    ATOT=ATOT+YC(I)
      YC(I)=(1.0-YC(I))*X(NBASE)
30    F(I)=(YD(I)-YC(I))/SQRT(YD(I))
      RETURN
C
C  ON THE FINAL CALL OF CALFUN PRINT THE PEAK VELOCITIES
C
25    CONTINUE
```

```
      PRINT 90                                                          00 1240
      PRINT*," "                                                        00 1250
      PRINT*," "                                                        00 1260
      PRINT*," THE PEAK VELOCITIES  ARE:"                               00 1270
      PRINT*," "                                                        00 1280
      PRINT 100,(V(I),I=1,4)                                            00 1290
      PRINT*,"  "                                                       00 1300
      PRINT*,"  "                                                       00 1310
      PRINT*,"THE AREA UNDER PEAKS 1 AND 5 OF ALPHA IRON IS: ",AAFE     00 1320
      PRINT*,"  THE TOTAL ALPHA IRON AREA IS TWICE THAT AREA."          00 1330
      PRINT*,"  "                                                       00 1340
      PRINT*,"  "                                                       00 1350
      PRINT*,"THE AREA UNDER THE ENTIRE CURVE IS: ",ATOT                00 1360
      PRINT*,"  "                                                       00 1370
      PRINT 90                                                          00 1380
   90 FORMAT(1H1)                                                       00 1390
  100 FORMAT(5X,7F10.4)                                                 00 1400
      RETURN                                                            00 1410
      END                                                               00 1420
```

APPENDIX D

GENFIT Instructions

This appendix contains instructions for using GENFIT with one of the three CALFUNs listed in Appendices A, B, and C. It is presented in two parts; the first explains the control cards and parameters used when running GENFIT, the second describes how to alter FIVECALF for different temperature runs.

Control Deck

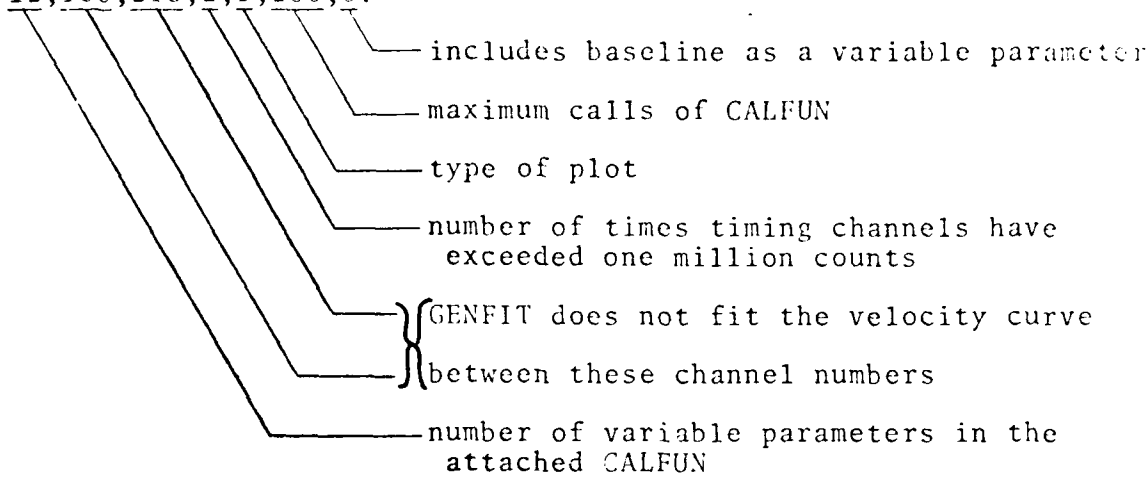
The following control deck precedes the data. It permits processing on the AFIT terminal only.

```
DEB,T300,CM120000,STCSB. M799999,DBELLER,4369.
ATTACH,A,GENFIT,MR=1
FIN,I=A,OPT=0,R=2,L=0.
ATTACH,COMPILE,GAUSSCALF,MR=1
FIN,I,OPT=0,R=2,L=0.
ATTACH,P,CCPLOT56X,ID=LIBRARY,SN=ASD.
LIBRARY,P.
LDSET,PRESET=ZERO
LGO.
7/8/9
```

The first two cards of the data deck are title cards. They are used to identify the material and run number, and the CALFUN used for processing the data. The third data card contains processing parameters as explained below.

FEB1(11/15)80. 611 K.
FIVECALI

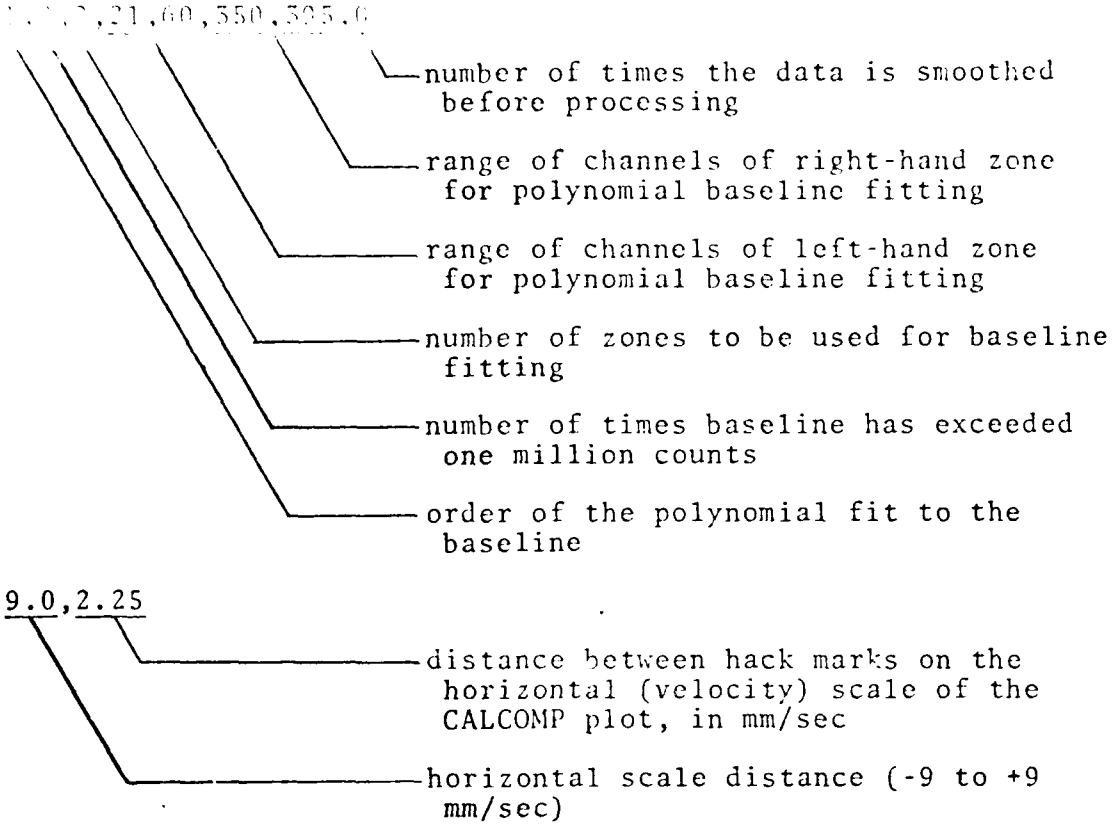
12,188,218,2,1,200,0.



The parameter cards then follow this card. In this case, there are twelve (as in above card). Two are given for example (format I6, open):

```
HFIELD    310.
   .
   .
   .
   .
   .
BASELN    52000
```

The Mossbauer data deck follows immediately after the parameter cards. There are forty data cards, in I3, 10I7 format. They contain the channel number of the first data entry on each card plus 10 channels of data. The final two cards contain more processing parameters.



The final card for processing Mossbauer data is the end-of-job card:

6/7/8/9 END OF JOB

Altering FIVECALF

FIVECALF must be changed for different temperature runs because the glass spectrum changes over a given temperature range. The average hyperfine field, the distribution of fields, the relative intensity of peak two to peak one, and the isomer shift and quadrupole split all vary with temperature. Altering FIVECALF is rather simple, however. Once GAUSSCALF has been run with a non-crystallized spectrum and the parameters

obtained, the necessary changes to FIVECALF can be made.

They are listed below by line number in Appendix B.

000220 change temperature to that which this copy of FIVECALF will be for

001460 change H=145. to the hyperfine field obtained with GAUSSCALF

001470 change S=-.200 to the isomer shift obtained

001480 change Q=-.005 to the quadrupole split obtained

001840 change B1(1) to the value obtained for the linewidth of peak one

001850 change B1(2) to the value obtained for the linewidth of peak two

001860 change B1(3) to the value obtained for the linewidth of peak three

001870 change B1(4) to the value obtained for the linewidth of peak four

001880 change B1(5) to the value obtained for the linewidth of peak five

001890 change B1(6) to the value obtained for the linewidth of peak six

001950 change 1.30 to the value of the ratio of peak two to peak one

001980 change 1.30 to the value of the ratio of peak two to peak one

APPENDIX E

Applications of Metallic Glasses

In this appendix some of the existing and proposed applications of the metallic glasses are described. The mechanical, electrical, and magnetic properties of some of the glasses make them suitable for many uses. Two commercial applications have existed since 1976. A woven fabric has been manufactured for use as magnetic shielding. It performs as well as $Fe_{80}Ni_{20}$ foil, and it has the advantage of high flexibility. The other current application of metallic glass is in magnetostrictive delay lines, which take advantage of the large magnetostriction of the metallic glasses and the high change in Young's modulus with applied magnetic field. Other uses are envisioned which take advantage of various combinations of magnetic "softness," mechanical hardness, and high electrical resistivity.

Due to the ease of reversing magnetic fields in the metallic glasses, power transformers with these materials in their cores would lose much less energy to heating. These glassy metals have been proposed for winding the cores of inversion transformers, current and pulse transformers, and magnetic amplifiers. They also are likely candidates for the "read" and "write" heads in magnetic tape recorders and disc

[illegible] systems. Their electrical resistance properties make them suitable for electrical resistors, low temperature heating wires, and resistance thermometers.

The various mechanical properties of the glassy metals make them useful for many other applications. Because of high tensile strength, some of the glasses might be used as reinforcing filaments in tires, transmission belts, or high pressure tubing; or as stress transducers in a multivibrator configuration. Their corrosion resistance makes them useful in underwater cables or biomaterials. The hardness and ability to be sharpened make some of the glassy metals suitable materials for manufacturing cutting devices.

Besides the above applications which are based on the macro-properties of the metallic glasses, there is at least one use based on their micro-structure: they have been proposed as the storage medium for magnetic "bubble" memory systems. Since the bubbles in the metallic glasses are one-fifth the size of those in synthetic garnet, the storage density would be 25 times greater. The vortices (important in superconductors) are 10 times smaller. If these can be used for storage, the information density could be 250 times greater than that now projected in synthetic garnet bubble memory systems.

All of the above applications can take advantage of two common properties of the metallic glasses: they are very easy to work with, due to their flexible-fiber form; and they are inexpensive to manufacture, due to the one-step production

proce[illegible], with no subsequent treatments necessary. A few of the authors who have researched the applications of the metallic glasses--and will probably continue--are: C.D. Graham, T. Egami, J.J. Gilman, P. Chaudhari, and F.E. Luborsky.

Vita

Denis Eugene Beller was born on November 22, 1950, in Okmulgee, Oklahoma, and is the son of Ernest E. and Ezma I. Beller. He graduated from Norte Del Rio High School in Sacramento, California in June 1968. He attended the University of Nevada and American River College before enlisting in the Air Force in 1971. In May 1976 he graduated with honors from the University of Colorado with a degree of Bachelor of Science in Chemical Engineering. Following graduation from Air Force Officer Training School, he served as a test engineer at the Air Force Rocket Propulsion Laboratory, Edwards AFB, California. Prior to attending the Air Force Institute of Technology, he served as a test section chief and senior test engineer at Edwards. He and his wife Judy have two sons.

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18 SUPPLEMENTARY NOTES

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19 KEY WORDS (Continue on reverse side if necessary and identify by block number)

Metallic Glasses
Aging Kinetics
$Fe_{80}B_{20}$
Crystallication Kinetics
Mossbauer Spectroscopy

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20 ABSTRACT (Continue on reverse side if necessary and identify by block number)

In this study, Mossbauer spectroscopy was used to examine thermal aging of two metallic glasses. $Fe_{80}B_{20}$ was isothermally annealed at 573, 604, 611, and 626 K and $Fe_{80}P_{6.5}C_{3.5}B_{10}$ was annealed at 614, 716, and 744 K. The activation energy of $Fe_{80}B_{20}$, determined from the growth of —>

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α-Fe crystals, was 0.256 ± 0.006 MJ/mole. The projected lifetime of this glass, based on the onset of crystallization, is 400 years. No quantitative data were obtained for $Fe_{80}P_{6.5}C_{3.5}B_{10}$; however, based on the higher temperature required for crystallization, it is expected to have a longer lifetime.

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